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AIC-167

## UTILIZATION OF ALFALFA

Mimeographed circular:

AIC-167 Alfalfa has promising chemurgic future. July, 1947. (See below under Processing).

### Constituents - Carotene

Carotene. I. Preliminary report on diphenylamine as a stabilizer for carotene. K. T. Williams, E. Bickoff, and W. Van Sandt. Science 97 (2508):96-98. Jan. 1943. A preliminary investigation was made of the stabilizing effect of about 100 substances of antioxidant character on carotene. Of these, diphenylamine had the greatest stabilizing effect.

Determination of carotene in vegetable oils without saponification. E. Bickoff and K. T. Williams. Indus. and Engin. Chem., Analyt. Ed. 15(4):266-268. Apr. 1943. A rapid method is described for the determination of carotene in vegetable oil solutions without saponification. The colored oxidation products of carotene that develop during storage are removed by this method. The carotene is separated from the other chromogens by passing a petroleum ether solution of the oil containing the mixed chromogens through a Tswett column of aluminum oxide, and eluting the carotene with 2 percent acetone in petroleum ether. The method also separates carotene from pigments extracted by petroleum ether (30°-60°C.) from dehydrated alfalfa, as well as from very large amounts of xanthophyll. The volume of sample and eluent must be controlled to ensure accurate separations of carotene by the column of aluminum oxide prepared as described.

Effect of antioxidants, individually and in combination, on stability of carotene in cottonseed oil. K. T. Williams, E. Bickoff, and B. Lowrimore. Oil and Soap 21(6):161-164. June, 1944. By an accelerated test, l-ascorbyl palmitate, alpha-tocopherol, hydroquinone, and phospholipids from cottonseed and soybean oil have been evaluated singly and in combinations, for their antioxidant effect on carotene added to mineral oil and cottonseed oil. Their effects in retarding formation of peroxides in the refined cottonseed oil were also determined.

Stability of carotene added to solid carriers. E. Bickoff and K. T. Williams. Indus. and Engin. Chem. 36(4):320-323. Apr., 1944. Addition of carotene to dry carriers in pellet form results in a very rapid loss of carotene. With protective measures, such as the addition of certain oils and of very small amounts of hydroquinone or diphenylamine as antioxidants, the retention of carotene is greatly improved. This suggests that pelleted mixtures containing extracted carotene may prove feasible as a supplementary feed.

Stabilization of carotene with nordihydroguaiaretic acid and other antioxidants. E. Bickoff, K. T. Williams, and M. Sparks. Oil and Soap 22(5):126-131. May, 1945. Edible oils fortified with carotene can be of value in supplementing our present supply of vitamin A. Fortification, however, necessitates stabilization, since carotene is readily lost by oxidation. Although added antioxidants have been successfully used to protect carotene in inert organic solvents, their value in edible oil solutions of carotene has not been well established. Relative values for the carotene-stabilizing effects on edible oil solutions of several antioxidants, alone and in various combinations, have been determined and are presented. One of the more promising is nordihydroguaiaretic acid. When used with phospholipid or citric acid, its effectiveness is enhanced. Addition of

alpha-tocopherol to oils containing only traces of this antioxidant causes a significant increase in carotene stability, and its sparing effect on carotene in vivo would further add to the desirability of its use in oil solutions of carotene. In refined cottonseed oil, carotene destruction occurred concurrently with the accumulation of peroxides. In the more saturated oils, carotene destruction occurred before much peroxide was detected in the oils.

Stability of carotene at elevated and room temperatures. E. Bickoff and K. T. Williams. *Oil and Soap* 23(3):65-68. March, 1946. Essentially an evaluation of elevated temperature tests used for carotene-stability studies in this Laboratory. Stability of carotene in edible oils containing various added antioxidants was determined by storage at 25°C. and in some cases 40°C. and compared with stability as determined by the accelerated tests at 75°C. Results obtained at 75°C. were in agreement with those obtained at 40° and 25°C. With most of the better antioxidants, protection at the lower temperature was better than that indicated by the rapid test. Solutions of carotene in edible oils, which ordinarily might lose a significant amount of carotene in a week or less at 25°C., can be protected by addition of antioxidants for periods well over a year without such special precautions as refrigeration or storage under nitrogen or in evacuated containers. Effectiveness of nordihydroguaiaretic acid as an antioxidant for carotene was confirmed by room-temperature storage tests.

#### Storage

Retention of carotene in alfalfa stored in atmospheres of low oxygen content. E. J. Hoffman, F. G. Lum, and A. L. Pitman. *Jour. Agr. Res.* 71(8):361-373. Oct., 1945. Storage of alfalfa meal at 40°C. in closed containers in contact with atmospheres of low oxygen content resulted in a high degree of carotene conservation. Conservation of carotene was due in large measure to reduction in the original oxygen concentration by oxidation of constituents of the alfalfa other than carotene in early stages of storage. The most effective conservation of carotene in alfalfa commercially by means of inert gas would be by the highest possible exclusion of air throughout the storage period. The permissible oxygen content of the storage atmosphere at the beginning was somewhat less than 3 percent.

#### Processing

Alfalfa has promising chemurgic future--a novel processing method. E. Bickoff, A. Bevenue, and K. T. Williams. *Chemurgic Digest* 6(14):213, 216-18. July 31, 1947. Freshly cut alfalfa was disintegrated to give a fiber-rich and a pigment-rich fraction. The juice was expressed from the pigment-rich fraction by the use of a hydraulic press. The press cake was dried and can be used as a feed ingredient. The juice was heated and the coagulated material removed by filtration. The coagulated material was 75 percent protein and rich in pigment. The juice was vacuum dried at low temperature. The press juice, without fractionation, has been spray dried to give a concentrated animal feed.

## EGG PROCESSING AND STORAGE

### Dehydration

Vitamin content of spray-dried whole egg. A. A. Klose, G. I. Jones, and H. L. Fevold. *Indus. and Engin. Chem.* 35(11):1203-1205, Nov., 1943. Spray drying of emulsified whole eggs, as practiced in most commercial drying units from which samples were obtained, was found to have no significant destructive action on vitamins, namely, vitamins A and D, thiamin, riboflavin, pantothenic and nicotinic acids. Vitamin A was found to be unstable during storage; after 9 months 60 percent was lost at 15°F., 75 at 70°, and 80 at 98.6°. Pantothenic and nicotinic acids were quite stable during 9 months. Only slight destruction of riboflavin occurred at 98.6°F. during 9 months and essentially none at lower temperatures. Thiamin was stable at 15°F. for 9 months; during the same period the losses were 46 percent at 70°F., and 50 at 98.6°F.

Determination of water in dry food materials. Application of the Fischer volumetric method. C. M. Johnson. *Indus. and Engin. Chem., Analyt. Ed.* 17(5):312-316, May, 1945. With the Fischer volumetric method it was possible to determine moisture content of starches, sugars, pectin, and spray-dried egg powder fairly rapidly and to get results that agreed with those obtained by the standard vacuum-oven technique.

Specific heats of dehydrated vegetables and egg powder. F. Stitt and E. K. Kennedy. *Food Res.* 10(5):426-436, Sept.-Oct., 1945. Mean specific heats of seven dehydrated vegetables and of egg powder were measured for temperature interval 27° to 66°C. Measurements between 0° and 27°C. were also made on four of these materials. Heat capacity was found to exceed the sum of the heat capacities of the equivalent quantities of dry solid and water by an amount which varies with moisture content in different ways for dehydrated carrots, eggs, and potatoes. These positive deviations from additivity are discussed with regard to origin, thermodynamic significance, and interpretation in terms of bound water content.

Vapor pressure of water adsorbed on dehydrated eggs. B. Makower, *Indus. and Engin. Chem.* 37(10):1018-1022, Oct., 1945. Equilibrium pressure of water vapor over spray-dried whole eggs was determined at six temperatures varying from 17.1° to 70°C. and at moisture contents ranging from 0.5 to 5.5 percent. The results agreed well with similar data on eggs dried from the frozen state. The results were analyzed by a graphical method and from this analysis was derived the isosteric heat of adsorption of water on dried eggs at various moisture levels. The ratio of isosteric heat to latent heat of condensation of water vapor increases from 1.1 to 2.1 as the moisture content decreases from 5.5 to 0.5 percent. It was also shown that the adsorption isotherms can be represented, within certain limits, by the Brunauer, Emmett, and Teller theory of adsorption of gases on solids.

Changes in stored dried eggs. Source of fluorescence. H. S. Olcott and H. J. Dutton. *Indus. and Engin. Chem.* 37(11):1119-1121, Nov., 1945. The fluorescence of 10 percent salt extracts of dried eggs was used as a simple criterion of palatability. This fluorescence, the source of which has heretofore not been recognized, appears to be due to the brown reaction products of glucose and the free amino groups of proteins. Similar fluorescing brown products also result from the interaction of simple amines and aldehydes and these can be used as model systems for the study of the glucose-protein reaction.

Changes in stored dried eggs. Role of phospholipids and aldehydes in discoloration. B. G. Edwards and H. J. Dutton. *Indus. and Engin. Chem.* 37(11):1121-1122, Nov., 1945. Dehydrated egg powders tend to acquire a brown color during storage. In a study of sources of this discoloration, an ether-soluble brown substance has been traced to the cephalin fraction of the phospholipids. Evidence obtained supports the hypothesis that this substance is the product of a reaction of an aldehyde with an amino constituent of the cephalin fraction.

Changes in stored dried eggs. Spectrophotometric and fluorometric measurement of changes in lipids. H. J. Dutton and B. G. Edwards. *Indus. and Engin. Chem.* 37(11):1123-1126, Nov., 1945. Changes in color of the lipid fraction of dehydrated egg during storage arise presumably from three chemical changes: destruction of naturally occurring carotinoid pigments, production of yellow-to-brown materials from unsaturated fatty acid groups, and development of brown products resulting, probably, from interaction of amine and aldehyde groups of lipids. While carotinoid destruction results in loss of color, the other two changes tend to darken the dried egg product. Changes that occur in the lipid fraction of dehydrated eggs during storage were studied by spectrophotometric and fluorometric techniques. The reaction of lipid amines with aldehydes, the destruction of carotinoids, and a process interpreted as polymerization of the unsaturated fatty acids were found to proceed faster at 98°F. than at 70°; little change was detected at 15°F. The fluorescing substance that develops in the fat of dehydrated eggs during storage was tentatively identified as the reaction product of lipid amines with aldehydes.

Determination of carotenoids and lipid amine-aldehyde products in dehydrated egg. H. J. Dutton and B. G. Edwards. *Indus. and Engin. Chem., Analyt. Ed.* 18(1):38-41, Jan., 1946. A combined spectrophotometric-fluorometric method is presented by which carotenoids and lipid amine-aldehyde products can be determined directly upon ether extract of egg powder without recourse to saponification and solvents partition. A spectrophotometric method for the determination of carotinoid content and a fluorometric method for the determination of ether-soluble brown products in powdered eggs are described. The development of brown lipid substances in stored egg powders introduced errors into the direct photometric measurement of carotenoids; however, these errors are minimized by the spectrophotometric procedure described. Both determinations can be performed upon a single ether extract of egg powder.

Factors influencing estimation of free fatty acids in dried egg powders. L. Kline and C. M. Johnson. *Indus. and Engin. Chem., Analyt. Ed.* 18(1):35-38, Jan., 1946. At the normal pH of dried whole egg, fatty acids are incompletely estimated by the A.O.A.C. procedure for determination of acidity of ether extract. In addition, the contribution of the extracted cephalin to the acidity of the ether extract may obscure significant percentage increases in the free fatty acidity of the egg powder. The necessity for drying the egg powder as required in the A.O.A.C. procedure is re-emphasized.

Determination of free fatty acids in dried egg powders. C. M. Johnson and L. Kline. *Indus. and Engin. Chem., Analyt. Ed.* 18(10):617-619, Oct., 1946. In the method described, egg powders are extracted with acetone, and traces of cephalin are removed with magnesium chloride before titration. The acidity of extracts of powders from which cephalin has been removed is not appreciably influenced by variations in moisture content of powder (at least within the range 0.3 to 6.5 percent). Errors caused by formation of nonextractable fatty acid-protein complex were studied. The fatty acids thus bound can be recovered completely from liquid egg emulsions only if the emulsion is adjusted to pH 4 before drying and extracting. Fatty acids which develop in dry egg powder during storage are not bound and can be completely extracted from the dried state by the proposed method.

Changes in color of dehydrated eggs during storage. H. J. Dutton and B. G. Edwards. *Indus. and Engin. Chem.* 38(3):347-350, March, 1946. Color changes from yellow to brown during storage of dehydrated eggs were studied spectrophotometrically by reflectance and absorption measurements. The influence of the simultaneous destruction of carotinoids and formation of brown aldehyde-amine substances upon reflectance of egg powders is discussed. The lipid amine-aldehyde reaction is dependent on moisture content, whereas rate of carotinoid destruction is independent of moisture content. Both reactions are accelerated by increasing temperature of storage. An approximately linear relation has been observed between negative logarithm of reflectance of residues after ether extraction and fluorescence of salt extracts of residues. This linearity confirms the conclusions previously drawn from experiments with model systems that the salt-extractable fluorescent compound, which has been used as an index of palatability, is the brown "glucose-protein" product.

Determination of surface area of dehydrated egg powder. T. M. Shaw, A. R. Vorkoeper, and J. K. Dyche. *Food Res.* 11(3):187-194, May-June, 1946. This paper reports the use of the low temperature-gas method and the permeability method for determination of surface areas of representative spray-dried and lyophilized egg powders. The surface areas found by the low temperature-gas adsorption method range from about 9.1 to 9.8 square meters per gram. Lyophilized powders have larger areas than spray-dried powders. The permeability method is shown to be unsatisfactory for accurate determination of surface areas of dehydrated egg powders because of anomalous dependence of surface area on the porosity in the permeability cell.

Dehydrated egg powders. Factors in palatability of stored powders. M. M. Boggs and H. L. Fevold. *Indus. and Engin. Chem.* 38(10):1075-1079, Oct., 1946. Effects of decreasing moisture content, gas packing, acidification, and acidification plus gas packing, on the shelf life of dehydrated egg powders during storage at 36.5°C. are reported. "Shelf-life" is defined as the time of storage during which the egg powders remain acceptable for consumption as scrambled eggs. Egg powders of lower moisture content retain palatability better than those containing more moisture. Packing in nitrogen or carbon dioxide increases shelf life two- and fourfold, respectively. Acidification of egg emulsion to pH 5.5 before drying resulted in a two- to threefold increase in shelf life over unacidified powders. Acidification plus carbon dioxide or nitrogen packing brings about the best retention of palatability during storage, shelf life being five to six times that of nonacidified air-packed egg powders. Acidified spray-dried powders retain good beating properties.

Dehydrated egg powders. Sources of off-flavors developed during storage. H.L. Fevold, B. E. Edwards, A. L. Dimick, and M. M. Boggs. Indus. and Engin. Chem. 38(10):1079-1082, Oct., 1946. Egg white does not contribute materially to loss of palatability during storage. Egg yolk deteriorates and imparts the characteristic "stored" flavor. Of the egg yolk components, the proteins, lipovitellin and livetin do not develop stored flavor and odor. The phospholipid fraction of lipids seems to be the source of substances imparting off-flavors and off-odors. The phospholipid-free lipids develop marked rancidity when stored separately, which does not occur in the presence of the phospholipids. It thus appears that phospholipids act as antioxidants, but in so doing undergo changes which impart off-flavors and off-odors. Salt-water fluorescence measurements of recombined whole egg powders show that the fluorescing substance arises in the egg white rather than in yolk. The reliability of this criterion for palatability determination is therefore questionable.

Dehydrated egg powders. Relation of lipid and salt-water fluorescence values to palatability. M. M. Boggs, H. J. Dutton, B. G. Edwards, and H. L. Fevold. Indus. and Engin. Chem. 38(10):1082-1084, Oct., 1946. Results of experiments to evaluate palatability of egg powders objectively. Salt-water fluorescence values for stored powders were found to correlate well with palatability scores for high-moisture (4 to 5 percent) powders, but the correlation was poor for powders below 2 percent moisture. Lipid fluorescence values, on the other hand, correlated well with palatability scores for high and low moisture powders during storage, and the correlation was also better for lipid fluorescence when all classes and grades of egg powders are considered. Because of these facts, and since the lipid-soluble fluorescing substance and the substance mainly responsible for loss of palatability both apparently originate in the phospholipid fraction, lipid fluorescence values are believed to be better criteria of palatability than salt-fluorescence values.

Resazurin reduction test and microbiology of egg powders prepared by lyophilization. D. J. Hirschmann and H. D. Lightbody. Food Res. 12(5):372-380, Sept.-Oct., 1947. The resazurin dye-reduction test was applied to uninoculated whole egg emulsions, to emulsions inoculated with Pseudomonas fluorescens, and to powdered products prepared by lyophilization. Although a general relationship was found between plate count and dye-reducing time, this relationship was disrupted by lyophilization and further by storage of powder. The dye-reducing action of emulsions of inoculated powders remained relatively high, although plate counts were reduced approximately 99 percent by drying and further decreased during storage. Dye-reducing action by uninoculated egg liquid and by emulsions of uninoculated powder was slow. Storage of uninoculated powder at 95°F. caused a slight increase in reducing action. The study suggests that sanitary history of certain foods not subjected to heat treatment in process of manufacture might be gauged by the resazurin test.

Effect of bacteria on quality of stored lyophilized egg powders. D. J. Hirschmann and H. D. Lightbody. Food Res. 12(5):381-392, Sept.-Oct., 1947. Whole egg liquids, uninoculated and inoculated with Pseudomonas fluorescens, were lyophilized and resulting powders stored at 95° and -20°F. Inoculated powders stored for 12 weeks at 95°F. showed marked increases in free fatty acid content, higher lipid fluorescence values, and increased resazurin color changes as compared to stored uninoculated powders. Inoculated powders stored at 95° and -20°F. showed lower pH and palatability values than corresponding uninoculated powders. The addition of Ps. fluorescens caused increase in salt-soluble fluorescence before storage, and increased lipid fluorescence during



storage at 95°F. over that of the corresponding uninoculated powders. Inoculated powders, to which resazurin was added before lyophilization, increased in rate of dye-color change during storage above that shown by uninoculated powders. It appears that deteriorative changes in inoculated stored egg powders are related to activity of nonreproducing bacteria or to cell components (presumably enzymes) that remain active after death of the organisms. The studies emphasize importance of plant sanitation and necessity of using egg liquids of high sanitary quality.

#### Characterization of Components (White)

Effect of acylating agents on the sulfhydryl groups of crystalline egg albumin. H. Fraenkel-Conrat, Jour. Biol. Chem. 152(2):385-389, Feb., 1944. At pH 5 to 6, phenyl isocyanate and carbon suboxide were found to react with thiol groups in preference to amino or phenolic groups of crystalline egg albumin. Ketene treatment caused more rapid acetylation of amino groups than of thiol groups. The thio esters formed by any of these reagents were hydrolyzed by alkali at room temperature, permitting almost complete recovery of protein thiol groups. Such reversible acylation of sulfhydryl groups was also demonstrated in model experiments with cysteine and glutathione.

The surface area of a crystalline egg albumin, T. M. Shaw, Jour. Chem. Physics 12(9):391-392, Sept., 1944. The adsorption of nitrogen at  $-183^{\circ}$  and water vapor at  $20^{\circ}\text{C}$ . was determined for crystalline egg albumin. The data presented in this paper indicate that water is able to penetrate into the polar regions of the crystallites and that nitrogen does not.

Isolation of lysozyme from egg white. G. Alderton, W. H. Ward, and H. L. Fevold, Jour. Biol. Chem. 157(1):43-58, Jan., 1945. The method depends on (a) adsorption of lysozyme on bentonite, (b) elution of inactive contaminating proteins from the clay by successive washings with phosphate buffer (pH 7 to 8) and 5 percent aqueous pyridine, and (c) elution of the active material with pyridine-sulfuric acid solution at pH 5.0. The eluate is dialyzed and dried in the frozen state. A white powder is obtained containing 85 to 90 percent of the lysozyme contained in the egg white. The product was crystallized, its isoelectric point and basic character were determined, and its homogeneity proved by electrophoretic measurements.

The relationship of lysozyme, biotin, and avidin. G. Alderton, J. C. Lewis, and H. L. Fevold, Science 101(2615):151-152, Feb., 1945. No evidence was obtained that either avidin or biotin is involved in the lytic activity generally ascribed to the lysozyme of egg white.

Optical and crystallographic properties of lysozyme chloride. F. T. Jones, Jour. Amer. Chem. Soc. 68(5):854-857, May, 1946. This paper reports the optical and crystallographic properties of single crystals of lysozyme chloride determined on air-dried and on wet crystals. The refractive indices have been found to vary with moisture content, but the birefringence remains constant.

Direct crystallization of lysozyme from white and some crystalline salts of lysozyme. G. Alderton and H. L. Fevold, Jour. Biol. Chem. 164(1):1-5, July, 1946. Lysozyme was crystallized directly from egg white by a method which markedly facilitates isolation in pure form. Lysozyme crystallized over the pH range of 3 to 11 from 5 percent sodium chloride solutions yields two distinct crystal forms, one below pH 7.0 and the other at basic reactions. The chloride, bromide, iodide, nitrate, and carbonate of lysozyme were prepared in crystalline form. A method of crystallizing lysozyme from egg white directly is described. Yields of 60 to 80 percent were obtained.

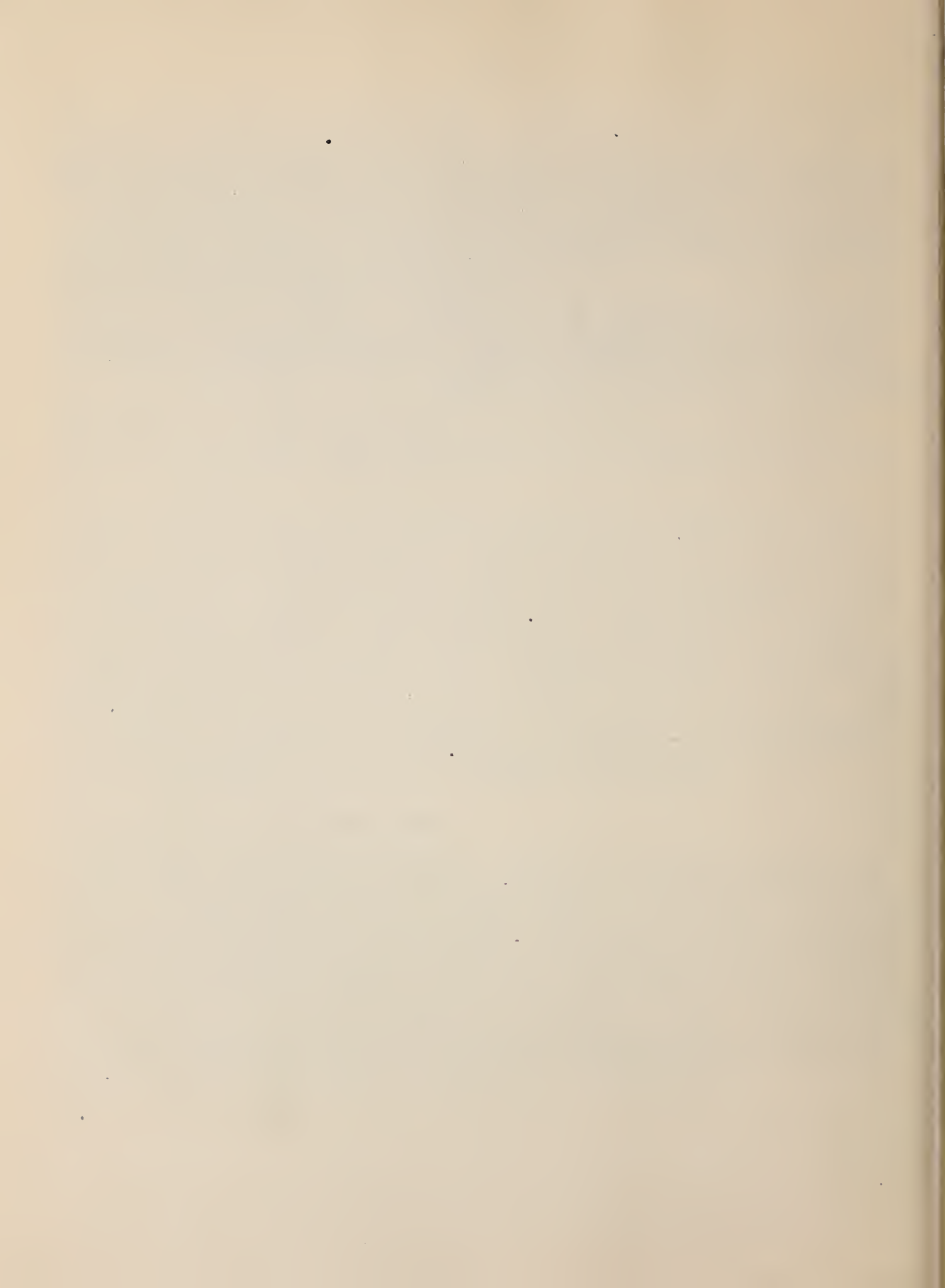
Identification of the bacteria-inhibiting, iron-binding protein of egg white as conalbumin. G. Alderton, W. H. Ward, and H. L. Fevold, Arch. Biochem. 11(1): 9-13, Sept., 1946. The authors purified the iron-binding antibiotic protein of egg white and identified it electrophoretically as 95 percent (or more) conalbumin. Approximately 80 percent of the conalbumin of egg white was isolated in this fraction. The antibacterial protein and conalbumin appear to be identical. Crystalline egg albumin, crystalline lysozyme and the globulin fraction of egg white are incapable of producing bacterial inhibition by iron deprivation.

Identification of the trypsin inhibitor of egg white with ovomucoid. H. Lineweaver and C. W. Murray, Jour. Biol. Chem. 171(2):565-581, Dec., 1947. Antitryptic activity was found in equal amounts in thin and thick white and was found quantitatively in the ovomucoid fraction prepared without heat treatment. Attempts to fractionate it further were unsuccessful and a limited electrophoresis study failed to reveal components other than ovomucoid. One molecule of ovomucoid (molecular weight, 29,000) caused about 50 percent inhibition of one molecule of trypsin. Heat-denatured ovomucoid had no antitryptic activity. Both native and denatured ovomucoid are soluble in water. Partial analysis revealed the following percentage composition: Nitrogen, 13.0; amino nitrogen, 0.7; acetyl, 4.5 to 5.2; sulfur, 2.2; cystine, 6.1; histidine, 3.2; tyrosine, 5 to 6; tryptophan, 0.3 or less; carbohydrate, 21.6 or more (as glucose); optical rotation,  $-56^{\circ}$  (sodium light  $20^{\circ}\text{C}.$ ); and SH (free or masked), none detectable.

#### Characterization of Components (Yolk)

Preparation of the egg yolk lipoprotein, lipovitellin. G. Alderton and H. L. Fevold, Arch. Biochem. 8(3):415-419, Dec., 1945. A convenient method for preparation of lipoprotein from egg yolk which consists of collecting crude lipoprotein by passing diluted egg yolk through a Sharples centrifuge and extracting precipitate with cold ether, is presented. Product thus obtained contains (in addition to lipovitellin) phosphorus-containing substances, which are removed by solution in 10 percent sodium chloride and precipitation by dialysis. Analytical data for final product agree well with published values. Approximately 18 percent of egg yolk solids was found to be lipovitellin; roughly 17 percent was isolated by the reported method. Lipoprotein contains approximately 16 to 18 percent of a phosphatide, which appeared to be largely lecithin.

Isolation of a new lipoprotein, lipovitellenin, from egg yolk. H. L. Fevold and A. Lausten, Arch. Biochem. 11(1):1-7, Sept., 1946. The new lipoprotein, which has been called lipovitellenin, contains 36-41 percent alcohol-extractable phospholipid as compared to 16-18 percent for the previously recognized lipoprotein, lipovitellin. Lipovitellenin dissolves to an opalescent solution in 10 percent NaCl but to a clear yellow solution in 10 percent NaCl saturated with ethyl ether. The new lipoprotein comprises roughly 40 percent of the total lipoprotein of egg yolk and 12 to 13 percent of the egg yolk solids. The protein component (vitellenin) of the lipoprotein is a phosphoprotein similar to vitellin in general solubility behavior. It contains, however, less than one-third as much phosphorus as does vitellin.



## FRUIT AND VEGETABLE BYPRODUCTS

### Subtilin

#### Mimeographed circulars:

AIC-106 Production, concentration, properties, and assay of the antibiotic, subtilin. Jan., 1946. A summary of work at the Western Regional Research Laboratory prior to date of issue.

AIC-168 Annotated bibliography of subtilin. By J. C. Lewis. Oct., 1947. This bibliography covers all published work on subtilin to date, from other institutions as well as the Western Regional Research Laboratory.

The following journal articles by Western Regional Research Laboratory authors are abstracted in AIC- 168 (listed above).

Utilization of asparagus juice in microbiological culture media. H. Humfeld and I. C. Feustel. Proc. Soc. Expt. Biol. and Med. 54 (2): 232-235. 1943.

Subtilin--An antibacterial product of Bacillus subtilis. E. F. Jansen and D. J. Hirschmann. Arch. Biochem. 4(3):297-309. July, 1944.

A method for purification of subtilin (abstract). K. P. Dimick, G. Alderton, H. D. Lightbody, and H. L. Fevold. Fed. Amer. Socs. Expt. Biol. Proc. 6(1):247-248. March, 1947.

Enhancement of subtilin activity by methylation (abstract). J. C. Lewis and E. F. Jansen. Fed. Amer. Socs. Expt. Biol. Proc. 6(1): 270. March, 1947.

The microbiological assay of subtilin. J. C. Lewis, E. M. Humphreys, P. A. Thompson, K. P. Dimick, R. G. Benedict, A. F. Langlykke, and H. D. Lightbody. (Authors Benedict and Langlykke, NRRL). Arch. Biochem. 14(3): 437-450. Aug., 1947.

Subtilin production in surface cultures. J. C. Lewis, R. E. Feeney, J. A. Garibaldi, H. D. Michener, D. J. Hirschmann, D. H. Traufler, A. F. Langlykke, H. D. Lightbody, J. J. Stubbs, and H. Humfeld. (Authors Traufler and Langlykke, NRRL). Arch. Biochem. 14(3):415-425. Aug., 1947.

Subtilin production in submerged culture. J. J. Stubbs, R. E. Feeney, J. C. Lewis, I. C. Feustel, H. D. Lightbody, and J. A. Garibaldi. Arch. Biochem. 14(3):427-435. Aug., 1947.

Zinc as an essential element for growth and subtilin formation by Bacillus subtilis. R. E. Feeney, H. D. Lightbody, and J. A. Garibaldi. Arch. Biochem. 15(1):13-17. Oct., 1947.

Purification and some properties of subtilin. K. P. Dimick, G. Alderton, J. C. Lewis, H. D. Lightbody, and H. L. Fevold. Arch. Biochem. 15(1):1-11, 1947.

Nutritional studies on the formation of subtilin by Bacillus subtilis in surface cultures (abstract). R. E. Feeney, E. M. Humphreys, H. D. Lightbody, and J. A. Garibaldi. Fed. Amer. Socs. Expt. Biol. Proc. 6(1):250. 1947.

The following articles are not listed in AIC-168:

A new laboratory fermentor for yeast production investigations. I. C. Feustel and H. Humfeld. Jour. Bact. 52(2):229-235. August., 1946. An improved laboratory fermentor for the culturing of yeast, employing mechanical agitation for the dispersion of air, with an operating capacity of 500 to 2,000 ml., is described. The results obtained in propagating Torulopsis utilis were markedly superior to those obtained with a previously described fermentor employing a porous disc for air dispersion. The new fermentor is recommended for use in yeast propagation investigations, and may be found suitable in such fermentations as the submerged culture production of antibiotics by bacteria and molds.

+An improved laboratory-scale fermentor for submerged culture investigations. H. Humfeld. Jour. Bact. 54(6):689-696. Dec., 1947. A fermentor with an improved agitation-aeration device and a specially designed mechanical foam breaker is described. The device is so designed as to pull in air at atmospheric pressure. The foam breaker consists essentially of a specially designed disk and cone, rotated at relatively high speed, which cause the foam to break under the centrifugal force generated under these conditions. Data are presented on a yeast (Torulopsis utilis) propagation run, which indicate a method by which the fermentor can be utilized in continuous operation processes after a constant yeast volume has been attained. The adaptability of the fermentor for the cultivation of Bacillus subtilis for subtilin production is indicated.

#### Other Antibiotics

A quantitative method for the determination of tyrothricin. K. P. Dimick. Jour. Biol. Chem. 149(2):387-393. Aug., 1943. A quantitative method, based upon the hemolytic action of tyrothricin, is described for the determination of tyrothricin in culture media. This method is accurate to within 5 percent, as determined by recovery experiments, and as little as 100 gamma of tyrothricin per ml. of culture can be measured. One ml. of culture is sufficient for test purposes. Isolation studies indicated that about 75 percent of the activity of a culture is obtained by the ordinary extraction procedures.

Modification of gramicidin through reaction with formaldehyde. J. C. Lewis, K. P. Dimick, I. C. Feustel, H. L. Fovold, H. S. Olcott, and H. Fraenkel-Conrat. Science 102(2646):274-275. Sept., 1945. The toxicity of tyrothricin and of its components, gramicidin and tyrocidine, has been a limiting factor in general applicability of these antibacterial agents in medicine. Treatment of tyrothricin with formaldehyde resulted in a loss of up to 50 percent of the antibiotic activity. The action of formaldehyde on gramicidin gave a similarly reduced hemolytic effect but the antibiotic activity, estimated with Staphylococcus aureus, was found to be unchanged. Preliminary tests with rats (intraperitoneal injections) indicated that the modified gramicidin was considerably less toxic than untreated gramicidin.

Production of tyrothricin in cultures of *Bacillus brevis*. J. C. Lewis, K. P. Dimick, and I. C. Feustel. *Indus. and Engin. Chem.* 37(19):996-1004. Oct., 1945. Yields in excess of 2 grams per liter were obtained in shallow layers of medium. Maximum yields were found after 10 or 16 days at about 35°C. with 11-mm. layers. Complex sources of nitrogen, such as Bacto tryptone, acid hydrolysate of casein, corn-steep liquor, tryptic digest of soybean meal, and press juice concentrates from waste asparagus butts, proved most suitable; relatively simple substances such as glutamic acid, asparagine, or ammonium sulfate plus citric or malic acid, proved less effective. About 3 to 5 percent of a fermentable carbon compound, such as glucose, mannitol, or glycerol, was necessary for best yields. Requirements for calcium, magnesium, and manganese were demonstrated.

Micro-Kjeldahl determination of nitrogen in gramicidin and tryptophan. Comparison of Gunning-Arnold-Dyer and Friedrich methods. L. M. White and G. E. Secor. *Indus. and Engin. Chem.* 18(7):457-458. July, 1946. Reports that mercuric oxide is a suitable catalyst for microdetermination of nitrogen in tryptophan (indole and amino nitrogen) and its acetyl and benzoyl derivatives. By its use recovery of nitrogen from the carboline ring is complete. With mercuric oxide as a catalyst, nitrogen can be determined in gramicidin and in the gramicidin-formaldehyde reaction product without the customary pretreatment with hydriodic acid.

Succinic acid derivatives of gramicidin and methylol gramicidin. H. S. Olcott, J. C. Lewis, K. P. Dimick, H. L. Fovold, and H. Fraenkel-Conrat. *Arch. Biochem.* 10(3):553-555. Aug., 1946. A reaction product of gramicidin and succinic acid with properties of possible therapeutic interest is reported.

Chemical derivatives of gramicidin. H. Fraenkel-Conrat, J. C. Lewis, K. P. Dimick, B. G. Edwards, H. C. Reitz, R. E. Ferrel, B. A. Brandon, and H. S. Olcott. *Proc. Soc. Expt. Biol. and Med.* 63(2):302-308. Nov., 1946. Derivatives obtained by reaction of indole or hydroxyl groups of gramicidin with numerous reagents are described.

The reaction of formaldehyde with proteins. IV. Participation of indole groups. Gramicidin. H. Fraenkel-Conrat, B. A. Brandon, and H. S. Olcott. *Jour. Biol. Chem.* 163(1):99-118. Apr., 1947. Gramicidin binds rapidly in alkaline solution an amount of formaldehyde equivalent to its tryptophane content. In acid solution the reaction proceeds only at high temperatures and formaldehyde concentrations. The formaldehyde adds as methylol groups probably to the nitrogen of the indole rings.

Antibiotic activity of the fatty-acid-like constituents of wheat bran. H. Hunfeld. *Jour. Bact.* 54(4):513-517. Oct., 1947. A fraction having antibiotic properties was extracted from wheat bran. This material has the characteristics of a fatty acid and forms a water-soluble potassium salt which has a comparatively high activity against *Staphylococcus aureus*, *Micrococcus conglomeratus*, and *Streptococcus faecalis*. It was inactive against *Escherichia coli*. When materials of plant or animal origin containing fats or fatty acid constituents are used in making

microbiological media, the possibility of these constituents having antibiotic activity, which might be confused with activity produced by microorganisms, should be given consideration.

Asparagus-Juice Medium. Yeast, Mushrooms

Mimeographed circular:

AIC-70 Process for production of asparagus-juice concentrate. Feb., 1945. Describes laboratory and pilot-plant studies, methods of production, processing costs.

Journal articles:

Relationship of iron nutrition to the synthesis of vitamins by Torulopsis utilis. J. C. Lewis. Arch. Biochem. 4(2):217-223. May, 1944. Attention has been centered on the production of yeast high in vitamin content from the press juices of waste and cull fruits. Of ten growth factors of the vitamin B complex studied, the synthesis of nine by Torulopsis utilis is significantly affected by the iron nutrition of this yeast. Iron deficiency is accompanied by increased rates of synthesis per gram of yeast for thiamin, riboflavin, nicotinic acid, pyridoxin and pyridoxin isobutels, and by decreased rates of synthesis for biotin, inositol, p-aminobenzoic acid, and Norit eluate factor isobutels. It is concluded that studies of mineral nutrition offer considerable promise in a search for factors affecting the microbiological syntheses of vitamins.

\*Vitamin synthesis by torula yeast. J. C. Lewis, J. J. Stubbs, and W. M. Noble. Arch. Biochem. 4(3):389-401. July, 1944. Torulopsis utilis grown in high yields on fruit juice substrates was assayed for members of the B group of vitamins. The vitamin content was found to be roughly comparable to that of bakers' yeasts, and certain other microorganisms. When the high yields of Torulopsis utilis obtainable from cheap substrates are considered, it appears that this yeast is an outstanding vitamin producer. Significant amounts of a number of the vitamins are also found in the culture liquors.

\*Fruit juices yield food yeast. J. J. Stubbs, W. M. Noble, and J. C. Lewis. Food Indus. 16(9):694-696. Sept., 1944. Synthesis of protein through the growth of yeast on sugars and simple inorganic materials is of widespread interest at present. Yeast protein, which may make up half or more of the yeast dry substance, is regarded as having high nutritive value, and the yeast substance is an excellent source of B vitamins. These factors establish the value of yeast as a component of animal and poultry feeds and as a food supplement for man. The article shows that fruit waste materials furnish suitable media for torula yeast propagation. The technique developed for bakers' yeast can be used. Raw material is cheap and plentiful and the product has high nutritive value.

Methionine deficiency in yeast protein. A. A. Klose and H. L. Fevold. Proc. Soc. Expt. Biol. and Med. 56(2):98-101. 1944. This paper reports that brewer's yeast and torula yeast grown on molasses, when either was



fed as the sole source of protein to growing rats, were found to be lacking in sufficient available methionine to produce growth equal to that elicited by an equivalent amount of casein. It appears that this fact is one limiting factor which must be considered in evaluating yeast protein as a replacement for animal proteins.

Nutritional value of yeast protein to the rat and the chick. A. A. Klose and H. L. Fevold. Jour. Nutrition 29(6):421-430. June, 1945. Brewers' yeast, torula yeast grown on molasses, and a torula yeast grown on prune contained an inadequate amount of methionine for optimum rate of growth in a comparative study with relatively complete proteins. This inadequacy could be corrected by the addition of methionine or methionine-rich protein to the diet. Cystine corrected the deficiency to a limited extent. Yeasts at relatively high levels and over limited periods did not appear to be toxic.

Spectrographic determination of calcium in microbiological culture media. E. J. Eastmond. Jour. Optical Soc. Amer. 38(1):57-60. Jan., 1946. One of a series of investigations undertaken at this Laboratory regarding the utility of asparagus-butt juice as a microbiological culture medium involved the effect of various metallic constituents on mold growth and bactericide yield. In order to follow changes in metallic composition of the medium as growth progresses, fairly rapid methods of analysis were considered desirable. This paper discusses the spectrographic determination of calcium.

Evaluation of torula yeast protein in the life cycle of the rat. A. A. Klose and H. L. Fevold. Arch. Biochem. 13(3):349-355. June, 1947. Rats were reared through four successive generations on purified diets with torula yeast as the sole source of protein. At a 20 percent level of crude protein, the yeast diet permitted suboptimum growth and successful matings and pregnancies, but was inadequate for the lactation period and resulted in death of the litters. Addition of methionine or cystine increased the growth rate and allowed a normal lactation period to occur. When pregnant rats were changed from an adequate stock diet to a yeast (torula or brewers') diet at parturition, supplementation with methionine or cystine greatly increased the weaning weight.

### Enzymes

(Abstracts of research articles on lysozyme are included in the list on Poultry Byproducts.)

Enzyme action in slaughtered meat animals. H. Lineweaver and T. L. Swenson. Proc. Ann. Meet. Amer. Inst. Refrig. pp. 94-103. 1941. During chilling, the major enzyme-catalyzed change is the formation of lactic acid. This process, generally complete in one day, is due to depletion of substrate, glycogen. The resulting lower pH, however, favors the deleterious rancidity (lipoxidase) changes, as well as the proteolytic (cathepsin) changes, which by no means reach completion during chilling. During aging chemical changes occur in the proteins of beef and lamb that apparently involve cathepsin and other enzymes acting on peptide

bonds or on amino acids. However, physical changes, which presumably are not connected with enzyme action, also occur. During storage changes due to enzymes are continuations of those occurring during aging.

Production of active and inactive catalase by Proteus vulgaris. T. L. Swenson and H. Humfeld. Jour. Agr. Res. 65(8):391-403. Oct., 1942. The existence of catalase in an inactive state has long been recognized. From data presented the following conclusions seem warranted: Proteus vulgaris produces catalase during all its growth, except in the lag phase. Proteus vulgaris produces two forms of catalase, active and activable. Kolmer's cholesterolized antigen, used as an activator, produced approximately a 4-fold increase in active catalase. The activable form cannot be extracted by acetone and therefore may be regarded as an integral part of the living cell--that is, a desmo enzyme. When the viable cells were destroyed, the activable catalase was either destroyed or could not be activated in the absence of viable cells.

Enzymic preparation and extraction of pectinic acids. H. S. Owens, R. M. McCready, and W. D. Maclay. Indus. and Engin. Chem. 36(10):936-938. Oct., 1944. Although pectin has been known for 120 years, only recently have serious attempts been made to prepare derivatives for other purposes than gelling agents. Activity has been stimulated by the fact that low methoxyl pectins are showing considerable promise in certain fields. The presence of a pectin esterase in citrus fruit peel has made it possible to develop an efficient procedure for the manufacture of a series of pectinic acids employing the enzyme in situ. The partially deesterified pectins extracted from the raw material form very viscous solutions indicative of long-chain molecules. Such materials, which differ from pectic materials now available commercially, may find wide usage wherever water-soluble, oil-repelling films are desired. Various salts differ in solubility characteristics and offer other possibilities for use. The conditions for the preparation of a series of pectic substances are described and several uses suggested.

\*Bacterial proteinase from waste asparagus butts. L. Kline, L. R. MacDonnell, and H. Lineweaver. Indus. and Engin. Chem. 36(12):1152-1158. Dec., 1944. The press juice from waste asparagus butts and trimmings has potential value as a constituent of industrial microbiological media. This paper describes the use of this juice for one process, the production of bacterial proteinase. Reproducible yields of proteinase were obtained on asparagus-juice media by control of pH and concentrations of calcium, carbohydrate, nitrogen, and phosphate, and by exercise of care with regard to culture technique. The culture filtrates, containing the proteolytic enzymes, were comparable with those obtained in commercial practice. Proteinaceous press cakes from oil seeds and carbohydrates, major raw material for commercial media, can be largely replaced by asparagus-butt juice.

The effect of cations on the activity of alfalfa pectinesterase. H. Lineweaver and G. A. Ballou. Arch. Biochem. 6(3):373-387. May, 1945. Presents data to show that activity of pectinesterase at pH 5.7 is about 30 times as great in the presence of 0.2M monovalent cations or 0.02M

divalent cations as in the absence of cations, but at pH 8.5 these concentrations of cations are practically without effect on the activity. The enzyme, only slightly inhibited by sodium pectate at pH 8.5, is markedly inhibited at pH 5.7 in the absence but not in the presence of cations. The enzyme-substrate dissociation constant at pH 5.7 is about the same (0.04 percent) at 0.025 and 0.2M Na ion. The enzyme is adsorbed on Celite near pH 5 and is eluted in dilute salt solution. The hypothesis correlating these observations is based on the assumptions, (a) that cations prevent inhibition by the pectin carboxyl groups by forming cation-carboxyl complexes, and (b) that the form of the enzyme existing in alkaline solutions has a decreased tendency to form an inactive complex with the carboxyl groups.

\*Properties of alfalfa pectinesterase (pectase). (Abstract.) H. Lineweaver and G. A. Ballou. Fed. Amer. Soc. Expt. Biol. Proc. 2:66. 1945. (See abstract above.)

The properties of orange pectinesterase. L. R. MacDonnell, E. F. Jansen, and H. Lineweaver. Arch. Biochem. 6(3):389-401. May, 1945. Orange pectinesterase was investigated because orange rind is a potentially cheap source of the enzyme, and because pectinesterase preparations from oranges, unlike those from tomatoes and some other sources, were found to be free of polygalacturonase. The data show that pectinesterase in citrus-fruit tissues is associated with solid particles. The increased activity caused by cations was dependent on pH of reaction mixture and varied from 5-fold at pH 7.5 to more than 100-fold at pH 5. Salt not only affected activity of orange pectinesterase but also affected extraction, adsorption, and elution and stability of the enzyme. The enzyme was inactivated slowly at 40°C. in the presence or absence of salt (pH 7.5) and lost about two-thirds of its activity in five minutes at 56°C. No polygalacturonase could be detected in extracts of orange flavedo by measurement of either reducing values or molecular weight.

Non-protectolytic, non-oxidative enzymes. H. Lineweaver and E. F. Jansen. Ann. Rev. Biochem. 14:69-90. 1945. Sixteen of the nonprotectolytic, non-oxidative enzymes are considered in this review of the year's work (1944) on these enzymes. In vitro properties are given special attention. Amidases, esterases, glycosidases, and miscellaneous enzymes, including lysozyme, are reported and advances noted.

Seasonal variation in the enzyme content of eleven varieties of carrots. H. J. Morris, C. A. Weast, and H. Lineweaver. The Bot. Gazette 107(3): 362-372. March, 1946. Inequalities of enzyme concentration in tissues of different species of plants, as well as in the same species at different stages of maturity, are well recognized. To study this phenomenon two harvests (2 months apart) were made of eleven varieties of carrots planted in August and in March. Carrots were of prime quality when harvested. Peeled portions were assayed for catalase, peroxidase, ascorbic-acid oxidase, and for those planted in March also for phosphatase. Activities per gram of peeled tissue were about the same as for the whole carrot root, except for peroxidase, which in samples tested was concentrated in the peel. Except for peroxidase, which varied little in average activity for all varieties highest activities were found for carrots

harvested in September, March, and January. Highest average enzyme activities were thus obtained under the most favorable growing conditions of temperature and photoperiod. Differences in average enzyme activity appear to reflect differences in growing condition rather than in age of the carrots (within limits of physiological age studied). Within single harvest differences in enzyme content attributable to variety ranged from 1.2-fold for phosphatase to 3.3-fold for ascorbic-acid oxidase, and averaged about 2-fold for the four enzymes studied. Ratios of the average enzyme contents for July (best growing condition) and January (poorest growing condition) harvests were 1.3 for peroxidase, 1.7 for catalase, and 5.5 for ascorbic-acid oxidase.

Citrus acetylsterase. E. F. Jansen, R. Jang, and L. R. MacDonnell. Arch. Biochem. 15(3):415-431. Dec., 1947. The properties of citrus acetylsterase, a previously uncharacterized type of plant enzyme, are presented. Data on extraction, distribution, stabilization, fractionation, and concentration are given. The activity of the enzyme as affected by salt, pH, and substrate concentration was determined. From specificity studies it was found that the enzyme best hydrolyzes esters of acetic acid and that it is not a lipase.

#### Carbohydrates, Particularly Pectin

\*The use of fibrous sodium pectate as a substitute for agar in bacteriological gels. R. M. McCready, H. S. Owens, and W. D. Maclay. Science 57 (2523):428. May, 1943. Importation of agar ceased during the war and a substitute was needed. This paper reports experimental use of fibrous sodium pectate in place of agar in bacteriological gels.

Preparation of d-galacturonic acid from pectin. E. Rietz and W. D. Maclay. Jour. Amer. Chem. Soc. 65(6):1242. June, 1943. Pectin is composed principally of polymers of d-galacturonic acid, and at present constitutes the most readily available source material for preparation of this compound. Preparation directly from pectin instead of from pectic acid is reported. A number of commercial pectins have been used, and yields ranging from 74 to 80 percent, based on uronic anhydride content of the pectin, are readily obtainable. Included were 285- and 300-grade apple pectins and 170-, 185-, and 200-grade citrus pectins with average yields of 78, 78, 80, 78, and 74 percent, respectively, of the uronic acid.

\*Pectin as an emulsifying agent. Comparative efficiencies of pectin, tragacanth, karaya, and acacia. H. Lotzker and W. D. Maclay. Indus. and Engin. Chem. 35(12):1294-1297. Dec., 1943. In this paper pectin as an emulsifying agent is compared with gums tragacanth, karaya, and acacia in a study of aqueous emulsions of olive, cottonseed, and mineral oils under various conditions of acidity, ratio of oil to water, and concentration of agent by measurement of changes with time in the specific interfacial surface of the dispersed oil, hydrogen-ion concentration, and viscosity.

Determination of soluble pectin and pectic acid by electrodeposition. K. T. Williams and C. M. Johnson. Indus. and Engin. Chem., Analyt. Ed. 15(1): 23-25. Jan., 1944. Investigations designed to develop new and extended uses for pectin made desirable an analytical method capable of determining small amounts of pectin. This paper describes a new approach to the determination of pectin, wherein solutions of pectin or pectates are deashed by the use of ion-exchange resins. The pectin or pectic acid is

electrolytically deposited at a platinum anode in a weighable form. The method requires less of the analyst's attention than does the calcium pectate method and, with partially purified solutions, gives results of the same order of accuracy. The method is especially applicable to amounts of pectin ranging between 5 and 50 mg.

\*Use of pectin in pharmaceutical pastes and ointments. W. D. Maclay, A. D. Shepherd, and H. Lotzkar. Jour. Amer. Pharm. Assoc. 33(4):113-116. April, 1944. Much attention has been given recently to the efficacy of various drugs and to the development of satisfactory washable gel- and emulsion-type bases in which to incorporate them. The use of pectin in this connection was studied, and this paper presents the following results: Tannic acid-pectin paste and an ointment base stabilized with pectin were prepared and their general stability over a period of four months studied. Various medicaments were incorporated into the paste and base, and some incompatibilities were observed. Freezing arrested changes in the paste and base. At room temperature the pastes and bases gradually became more acidic but were stable. At 55°C. they became discolored and gelled after five months' storage. The paste and base fulfill the requirements specified by the National Research Council except when stored at 55°C. A comparison between the pectin paste and the tragacanth paste indicated that pectin and tragacanth have the same advantages and disadvantages.

Spectrographic determination of lead in pectinous materials. C. R. Jeppesen, E. J. Eastmond, and H. G. Logan. Jour. Optical Soc. Amer. 34(6):313-318, June, 1944. (Abstract, Bul. Amer. Phys. Soc. 18(4):6; Phys. Rev. 64(5-6):188, 1943.) To follow the amounts of lead present in various portions of pectinous solutions taken during the process of purification of the pomace extract and to determine the amount in the purified solid pectin product, a spectrographic method was used for detection and measurement of these trace amounts of lead. Describes preparation of standards and samples, and outlines the spectrographic procedure.

\*Viscosities of pectin solutions. H. S. Owens, H. Lotzkar, R. C. Merrill, and M. Petersen. Jour. Amer. Chem. Soc. 66(7):1178-1182. July, 1944. The relative viscosity of pectin solutions varies with concentration in a manner similar to that of other ionizable hydrophilic colloids and certain salts. A tentative hypothesis is proposed to explain the results obtained.

Errors in the Zeisel methoxyl values for pectin due to retained alcohol. E. F. Jansen, S. W. Waisbrot, and E. Rietz. Indus. and Engin. Chem. 16(8): 523-526. Aug., 1944. Errors due to retained alcohol should be guarded against, particularly in studies of pectin structure, since most frequently pectin and its derivatives are precipitated from solution with ethanol, and ordinary drying technique does not remove all the alcohol. Data show that retained ethanol causes the methoxyl content of pectins, as measured by the Zeisel method, to be as much as 20 percent higher than saponification values. This ethanol cannot be removed by the usual drying techniques but can be removed by humidification followed by drying. Acetone rather than ethanol precipitation results in good agreement between the two methods. Retained isopropyl alcohol can be removed by drying at 100°C.

Xylitol esters of fatty acids. J. F. Carson and W. D. Maclay. Jour. Amer. Chem. Soc. 66(9):1609-1610. Sept., 1944. A series of fatty acid

esters of the pentahydric alcohol, xylitol, was prepared in connection with a study of plasticizers. The physical and chemical constants of the xylitol esters were studied and data are presented.

Alkali-hydrolyzed pectins are potential industrial products. R. M. McCready, H. S. Owens, and W. D. MacLay. Food Indus.: I. 16(10):69-71. Oct., 1944; II. 16(11):92-94. Nov., 1944. The characteristics of low-methoxyl pectins are comparable in some respects to gelatin and starches. Gels prepared with low-methoxyl pectin are palatable, smooth and firm, without rubbery or pasty consistency. Their preparation requires little time. Only a very short period of heating is required to dissolve the pectinic acid, and gelation occurs rapidly on cooling without refrigeration. The fact that during preparation these pectin products remain very fluid offers an additional advantage. This pectinic acid is versatile; its properties are such that it gels with milk and also with fruit and vegetable products. Low-methoxyl pectins require a different method of evaluation. Their methoxyl content and viscosity make them suitable for use in low-sugar jellies having the correct acidity and concentration of calcium ions.

The acylation of pectin. J. F. Carson and W. D. MacLay. Jour. Amer. Chem. Soc. 67(5):787-789. May, 1945. This paper describes a technique for the esterification of pectin. Pectin diacetate, dipropionate and dibutyrate were prepared by esterification of citrus pectin with the acid anhydride in pyridine. Lauryl, myristoyl, palmitoyl, and benzoyl esters of citrus pectin were prepared by esterification of pectin with the corresponding acid chloride in pyridine; the degree of esterification attained varied from 1.2 to 1.6 acid groups per anhydrogalacturonic acid unit.

Oriented fibers of sodium pectate. K. J. Palmer and H. Lotzkar. Jour. Amer. Chem. Soc. 67(5):883-884. May, 1945. Describes a method whereby well-oriented sodium pectate fibers can be made.

Acceleration by electrolytes of alkali de-esterification of pectin. H. Lineweaver. Jour. Amer. Chem. Soc. 67(8):1292-1293. Aug., 1945. The effect of sodium chloride, calcium chloride, and magnesium chloride on rate of alkaline de-esterification of pectin in aqueous solution is reported. These electrolytes, and in particular cations, have been shown to accelerate the rate of alkaline de-esterification of pectin as much as 400 percent. This acceleration contrasts with the small effect of electrolytes on rate of hydrolysis of simple esters. The results indicate that negatively charged carboxyl groups of pectin are close enough to the ester bonds to interact with the hydroxyl ions as they approach the ester bonds; salt decreases the interaction (repulsion). As has been found for the primary salt effect in other systems, the acceleration of de-esterification of pectin by salt reflects largely a change in the entropy rather than energy of activation.

1,4-anhydro-D,L-xylitol. J. F. Carson and W. D. MacLay, Jour. Amer. Chem. Soc. 67(10):1802-1810. Oct., 1945. Xylitol was dehydrated with benzene-sulfonic acid or sulfuric acid to yield a mixture of dehydration products from which a pure anhydroxylitol has been isolated in crystalline form. Anhydroxylitol was characterized by the preparation of three crystalline derivatives, the tribenzoate, tricarbonylate, and the monotrityl

diacetate. By oxidation with periodic acid and sodium metaperiodate, anhydroxyxylitol is shown to contain a 1,4 oxygen ring and is designated as 1,4-anhydro-D,L-xylitol.

Influence of methoxyl content of pectic substances on the action of polygalacturonase. E. F. Jansen and L. R. MacDonnell. Arch. Biochem. 8(1):97-112. Oct., 1945. Data show that pectinase complex hydrolyzed pectic acid (prepared with either pectinesterase or alkali) at an initial rate 17 times faster than it hydrolyzed pectin. Only the de-esterified portions of pectic substances were susceptible to the action of polygalacturonase. Extent of hydrolysis of pectic substances was a function of degree of de-esterification. Enzymic hydrolysis of 2 percent of the glycoside bonds of any of the substrates caused 50 percent of the total viscosity change. Hydrolysis of pectic acid by polygalacturonase goes through two stages: an initial rapid stage which extends up to approximately 45 to 50 percent hydrolysis of the glycoside bonds, followed by a slow stage in which the residual bonds are hydrolyzed. The first stage was 15 times as rapid as the second. The "half-hydrolyzed" material was not inhibitory to the hydrolysis of pectic acid. The Michaelis-Menten dissociation constant,  $K_m$ , for the first stage was less than 0.03 percent and for the second, greater than 1.0 percent. These observations suggest that the second stage was the hydrolysis of digalacturonic acid.

Simultaneous actions of polygalacturonase and pectinesterase on pectin. E. F. Jansen, L. R. MacDonnell, and R. Jang. Arch. Biochem. 8(1):113-118. Oct., 1945. Polygalacturonase, acting simultaneously with pectinesterase on pectin at pH 4.0, had a favorable effect on rate of de-esterification and on maintenance of rate, as compared with action of pectinesterase alone. This favorable effect was probably due to removal, by glycosidic hydrolysis, of pectic acid, which inhibits pectinesterase. In sufficiently high concentrations pectinesterase enhanced action of polygalacturonase on pectin to an extent that approached the rate observed on pectic acid. Higher concentrations of crude pectinesterase were inhibitory, probably because of the inert protein accompanying the esterase. No inhibition was observed with higher concentrations of purified pectinesterase. Pectinesterase of plant origin can be used to fortify the pectinase complex.

Configuration of the pyranose rings in polysaccharides. K. J. Palmer and M. B. Hartzog. Jour. Amer. Chem. Soc. 67(10):1865-1866. Oct., 1945. A comparison of the fiber identity periods determined by X-ray analysis of sodium alginate, alginic acid, sodium pectate, pectic acid, cellulose, soda cellulose, and soda cellulose II shows that its identity periods fall into four classes. This result is shown to be compatible with the assumption that the pyranose rings occur in either one of the two possible trans forms and that the chains have either two-fold or three-fold screw symmetry.

Influence of method of de-esterification on the viscosity and acid behavior of pectinic acid solutions. I. R. Schultz, R. Lobak, H. S. Owens, and W. D. MacLay. Jour. Phys. Chem. 49(6):554-563. Nov., 1945.

Considerable interest has developed in pectinic acids (colloidal polygalacturonides, partly esterified with methyl alcohol) that have lower ester contents than those used commercially for high-solidus gels. Viscosity-pH curves for high-methoxyl pectinic acids exhibited maxima near pH 6, while the low-methoxyl acids exhibited maxima near pH 6 and minima near pH 4. Under the conditions used, the transition from characteristics of high-methoxyl to those of low-methoxyl pectinic acids occurs at methoxyl content above 8 percent for esterase-deesterified pectinic acids and near 6 percent for those deesterified by acid or alkali. Pectinic acids deesterified by citrus pectinesterase are weaker than those of the same methoxyl content prepared with alkali. An hypothesis based on evidence that the esterase deesterifies portions of the galacturonide chain, while acid or alkali acts in a random manner, is proposed to explain the differences observed in behavior.

The thermal degradation of pectin. R. C. Merrill and M. Weeks. Jour. Amer. Chem. Soc. 67(12):2244-2247. Dec., 1945. The most obvious change when a solution of pectin is heated is the rapid irreversible decrease in viscosity, denoted in this paper by the term degradation. The large initial decrease in viscosity on heating is due mainly to the breaking of primary chemical valence bonds rather than to the destruction of a secondary aggregate. This is shown by the irreversibility of degradation, the absence of a pronounced effect of urea and temperature (up to 50°) on the viscosity of dilute pectin solutions, the fact that loss in relative viscosity is closely associated with a decrease in intrinsic viscosity of the pectin, and the magnitude of the value for the activation energy of the loss in viscosity with heating time (28,000 ± 6,000 cal./mole).

An X-ray diffraction investigation of sodium pectate. K. J. Palmer and M. R. Hartzog. Jour. Amer. Chem. Soc. 67(12):2122-2127. Dec., 1945. Interpretation of results indicates that the galacturonide chain has the configuration of a three-fold screw axis, and that the chains are arranged in closest packing. The structure has pseudo-hexagonal symmetry. The identity period in the fiber direction is 13.1 Å. This fiber identity period is somewhat less than the value found for some cellulose derivatives in which the chain has the configuration of a three-fold screw axis. This difference in identity periods is discussed in terms of molecular models. On desorption, sodium pectate in equilibrium with air at a relative humidity of 40 percent contains 18 percent water. A considerable portion of this water is located in the crystalline portion of the material. The non-uronide material (18 percent) is shown to have no detectable influence on the X-ray pattern.

Kinetics of the deesterification of pectin. R. C. Merrill and M. Weeks. Jour. Phys. Chem. 50(2):75-87. March, 1946. Summarizes work on rates of deesterification of one-percent solutions of purified citrus and apple pectins and of a commercial citrus pectin at 70°, 80°, 90°, and 100°C., and on influence of added acid, base, and salts. The deesterification is a first-order reaction with respect to pectin concentration and is catalyzed by acids and bases. The rate of deesterification is a minimum at approximately the natural pH of 4. Addition of most neutral salts to pectin solutions lowers the pH and increases the deesterification rate.



The influence of added salts on deesterification rate is due mainly to their effect on pH, but some specific ion effects are found. Effective ionic strengths of pectin solutions appear to be of the same order of magnitude as solutions of uni-univalent electrolytes of the same normality.

Effect of salts on the viscosity of pectinic acid solutions. H. Lotzkar, T. H. Schultz, H. S. Owens, and W. D. Maclay. Jour. Phys. Chem. 50(3): 200-210. May, 1946. This paper presents data on viscosity of pectinic acids in solution of salts which are encountered in various phases of pectin technology. Explanations are offered, wherever possible, for the behaviors observed.

Determination of uronic acids. R. M. McCready, H. A. Swenson, and W. D. Maclay. Indus. and Engin. Chem., Analyt. Ed. 18(5):290-291. May, 1946. The objective of this work was to devise an effective procedure for determining uronide content of pectins and other polyuronide substances in an appreciably shorter time than that required to carry out this type of analysis by currently recommended modifications of the Lefevre-Tollens method. Theoretical quantities of carbon dioxide were obtained for all uronic acids and their derivatives by heating the sample with 19 percent hydrochloric acid in an oil bath maintained at 145°C. for 1.5 to 2 hours. The apparatus is simple, compact, easy to construct, and offers a substantial saving of space and time. Analytical results on alginic acid were higher and more reproducible than those obtained by accepted methods.

The acylation of polyuronides with formamide as a dispersing agent. J. F. Carson and W. D. Maclay. Jour. Amer. Chem. Soc. 68(6):1015-1017. June, 1946. A new technique is described for acylation of polyuronides, in which formamide is used as a solvent or gelatinizing agent prior to esterification with pyridine and an acid anhydride.

Effect of methoxyl content of pectin on the properties of high-solids gels. H. S. Owens and W. D. Maclay. Jour. Colloid Science 1(4):313-326. July, 1946. Reports an examination of pectin gels, prepared under different conditions, with respect to modulus of rigidity, modulus of elasticity under compression, and breaking limit. Comparison of a high-solids gel prepared from rapid-set pectin with a high-solids gel prepared from slow-set pectin, partly deesterified by alkali or acid, was made at different pH values and in presence of calcium ion. A torsion-type instrument (rigidometer) was employed to obtain absolute measurements of modulus of rigidity, a sagometer was used to measure modulus of elasticity in compression, and a Tarr-Baker plunger-type instrument served as the tester for information on breaking limit. It was found that modulus of rigidity of pectin gels is related to molecular weight of pectin and that modulus of elasticity in compression can be related to that in shear if a Poisson ratio of 0.5 is assumed when small deformations are observed.

Shape and size of pectinic acid molecules deduced from viscometric measurements. H. S. Owens, H. Lotzkar, T. H. Schultz, and W. D. Maclay. Jour. Amer. Chem. Soc. 68(8):623-632. Aug., 1946. Reports viscosity behavior of various pectins in 0.155M sodium chloride solution studied under different conditions of pH and temperature. Intrinsic viscosity values were found to decrease with rise in temperature and to be essentially independent of pH and probably independent of the methoxyl content.

The intrinsic viscosity values for samples used have been calculated and related to the physical features of pectinic acid molecules. Length-to-diameter ratios calculated by means of Simha's equation varied from 53 to 165. Assuming a width of 10 A, the length of molecules varied from 530 to 1650 A. Molecular weights ranged from  $2.3 \times 10^4$  to  $7.0 \times 10^4$ . The results indicate that pectin has a rigid, rod-like structure in aqueous salt solutions. Osmotic pressure measurements were used to calculate number average molecular weights, which varied from  $1.8 \times 10^4$  to  $3.9 \times 10^4$ . Lack of agreement between these molecular weights and viscosity and weight average molecular weights is believed to be due to the heterogeneity of samples.

Esterification of galacturonic acid and polyuronides with methanol-hydrogen chloride. E. F. Jansen and R. Jang. Jour. Amer. Chem. Soc. 68(8):1475-1477. Aug., 1946. Concerns relative rates of glycoside formation and esterification at  $0^\circ$  and at  $24-25^\circ$  of galacturonic acid in absolute methanol containing various concentrations of dry hydrogen chloride. Esterification of galacturonic acid in methanol containing small concentrations of hydrogen chloride (0.005 to 0.1N) proceeded at a rate at least 25 times more rapid at  $25^\circ$  and 55 times more rapid at  $0^\circ$  than did glycoside formation. Both reactions were first order to at least 50 percent conversion and the energies of activation for esterification and glycoside formation reactions were  $14,000 \pm 700$  and  $21,000 \pm 1,000$  cal., respectively. Methyl alpha-D-galacturonate was isolated in good yields from such reaction mixtures. Alginic acid was partially esterified by a similar procedure. Pectic acid under the same conditions was only very slowly esterified.

Acidic isolation of low-ester pectinic acids. R. M. McCready, H. S. Owens, A. B. Shepherd, and W. D. Mackay. Indus. and Engin. Chem. 38(12): 1254-1265. Dec., 1946. An acidic method for isolating pectinic acids of low methyl ester content which requires neither concentration of pectinic acid solution nor use of organic precipitating agents is shown to be effective. The various factors affecting precipitability of this type of material are evaluated and a recommended procedure of isolation is presented.

\*An x-ray diffraction investigation of pectinic and pectic acids. K. J. Palmer, R. C. Merrill, H. S. Owens, and H. Ballantyne. Jour. Phys. and Colloid Chemistry. 51(2):710-720. May, 1947. X-ray photographs have been taken of pectinic and pectic acids in the form of both powders and fibers, and their interplanar spacings have been recorded. The variation in X-ray spacings with methoxyl content has been determined. A fiber identity period of about 13 A has been found for the polygalacturonide chain in all pectinic and pectic acids so far investigated. The inter-chain separation increases from approximately 6.6 A to 6.9 A when the methoxyl content varies from 0 to 11 percent. The sign of intrinsic birefringence of pectinic and pectic acid fibers is negative. This is in contrast to fibers of alginic acid and sodium alginate, which are positive. A structural reason for the negative sign of birefringence is suggested.

\*X-ray and moisture equilibrium investigation of sodium pectate. K. J. Palmer, T. M. Shaw, and M. Ballantyne. Jour. Polymer Science 2(3):318-328. June, 1947. Variation of X-ray reflection related to interchain separation in sodium pectate has been determined as a function of moisture content. The crystalline regions in sodium pectate have been found to sorb about 24 percent water (dry basis) at a relative humidity of 95 percent at 25°C. The sample as a whole under these conditions has a water content of 64 percent. At high water contents a large difference in water content must exist between crystalline and amorphous regions of sodium pectate. A marked change in degree of crystallinity occurs, as determined by number and sharpness of x-ray reflections at an average water content of about 6 percent on desorption and on sorption at an average water content of about 14 percent.

\*Use of polymetaphosphates and polyphosphates in the extraction of pectin and pectinic acids from citrus peel. R. M. McCready, A. D. Shepherd, and W. D. Maclay. Fruit Prod. Jour. and Amer. Food Mfr. 27(2):36-39. Oct., 1947. A study of action of polymetaphosphates and polyphosphates when used as aids in the extraction of pectin from citrus peel revealed the following advantages: (1) they permit use of a higher pH in obtaining maximum jelly-unit yield and (2) they are very effective in extraction of enzymatically de-esterified pectinic acids. Disadvantages are increased costs of chemicals and of filtration.

#### Patents

Method of extracting pectinous materials. No. 2,375,376. W. D. Maclay and J. P. Nielsen. Patented May 8, 1945. Improved method of extracting pectin from plant materials such as citrus peel, sugar beets, etc. which involves adding an alkali metaphosphate or alkali polyphosphate to the extracting solution. Process allows complete extraction in short period of time.

Method for accelerating the alkaline de-esterification of pectin. No. 2,386,323. H. Lineweaver and R. M. McCready. Patented Oct. 9, 1945. Improvement in the alkaline de-esterification of pectin to produce pectinic acids wherein a salt, such as sodium chloride, is added to the reaction mixture. The reaction is accelerated by addition of the salt. Invention permits use of mild alkali whereby degradation of pectin is avoided.

#### Fats and Oils

\*Minor oil-bearing crops of the United States. E. B. Kester and G. R. Van Atta. Oil and Soap 19(7):119-125. July, 1942. Discusses the subject of lesser known and little used vegetable oils, which may or may not be byproducts of crops grown in considerable abundance.

Glycidyl esters of aliphatic acids. E. B. Kester, C. J. Gaiser, and M. E. Lazar. Jour. Organic Chem. 8(6):550-556. Nov., 1943. Preparation of glycidyl esters of lauric, myristic, palmitic, stearic, oleic, and sebacic acids, and of the mixed fatty acids of babassu oil, castor oil, walnut oil, soybean oil, and rosin, by reacting anhydrous alkali salts of these acids with epichlorohydrin, is described. Included also are a

preparation of beta-methylglycidyl myristate from beta-methylepicichlorohydrin, and glycidyl palmitate and glycidyl sebacate by reacting acid chlorides with glycidol. Physical constants of the products are reported. The glycidyl esters are potential plasticizers and modifiers of vinyl resins.

Determining the mechanical stability of emulsions--a rapid quantitative method. R. G. Merrill, Jr. *Indus. and Eng. Chem., Analyt. Ed.* 15(12): 743-746. Dec., 1943. Reports development of method for determining the resistance of emulsions to breaking under mechanical stress of centrifugal force. This method involves measuring rate of separation of internal phase under a constant centrifugal force. The reciprocal of the initial rate of separation at a constant centrifuge speed has been taken as a quantitative index of mechanical stability of the emulsion. The method has been applied to both water-in-oil and oil-in-water emulsions stabilized by lecithin, soaps, and vegetable gums. It gives results in a few hours apparently comparable to those obtained by more tedious methods involving other factors and requiring measurements over many months. Definite effects of age of emulsion on mechanical stability as determined by this method have been found in the case of soap and saponin-stabilized emulsions.

Valencia orange-seed oil. G. R. Van Atta and W. C. Dietrich. *Oil and Soap* 21(1):19-22. Jan., 1944. Expansion in the citrus industry in recent years calls attention to citrus seeds as a potential though very minor oil source. California Valencia orange seeds contain a fairly high percentage of oil, the recovery of which by conventional methods of pressing appears to present no unusual problems. When refined, bleached, and deodorized, the oil would be quite acceptable as a food oil. It contains a very small proportion of linolenic acid but is otherwise similar to other seed oils of like iodine value.

Foaming properties of soap solutions. R. G. Merrill, Jr., and F. T. Moffett. *Oil and Soap* 21(6):170-175. June, 1944. Soaps and detergents are often appraised by the volume and stability of the suds produced. This paper reports results, obtained by the "liquid drainage" method and a modified "foam time" method, on the stability of foams from solutions of soaps and detergents. The two methods gave qualitatively equivalent results. Rates of drainage of liquid from soap and detergent foams were not proportional to the volumes of liquid in the foam. The stabilities of foams from 0.1 percent solutions of soaps of most of the natural fats and oils tested were much greater than those of foams from single soaps that were sufficiently soluble to form solutions of this concentration. The foam time of a mixture of equal volumes of two 0.1 percent solutions was not equal to the average of the foam times of the separate solutions. A number of electrolytes, organic liquids, pectic materials, and vegetable gums greatly increased foam stability. Data on 0.03 percent sodium tallow and palm oil soap solutions indicated a maximum foam time at a pH of about 10.6. Increasing the concentrations of ten soaps from 0.05 to 0.1 percent more than doubled the foam time.

Absorption analysis of colorless compounds: Method and application to the resolution of stearic and oleic acids. H. J. Dutton. *Jour. Phys. Chem.* 48(4):179-186. July, 1944. This paper presents a method for the

adsorption analysis of colorless compounds. The method employs a highly sensitive differential refractometer modified for measurement of changes in the refractive index of percolates from adsorption columns during continuous flow. The resolution of stearic and oleic acids has been investigated as an example of the application of this general method. Advantages and limitations of the method are discussed.

A modified Kreis test suitable for photocalorimetry. M. F. Pool and A. N. Prater. Oil and Soap 22(9):215-216. Sept., 1945. Because of its sensitivity and simplicity, the Kreis test is extensively used to detect oxidative deterioration in fats and oils. While the results are not a definitive index of rancidity, the information obtained is a highly useful supplement to other evidence. A sensitive form of the Kreis test for rancidity in fats and oils is described. The reaction occurs in a one-phase system which is suitable for direct photometric measurement. The procedure is convenient and rapid and yields reproducible results.

Tris-(hydroxymethyl)-phosphine oxide trilaurate. D. F. Houston. Jour. Amer. Chem. Soc. 68(5):914. May, 1946. Tetra-(hydroxymethyl)-phosphonium chloride formed by the action of phosphine on a solution of hydrogen chloride and formaldehyde was converted to tris-(hydroxymethyl)-phosphine oxide by means of barium carbonate. The crystallized product is soluble readily in benzene or chloroform, moderately in ether, and slightly in petroleum ether or methanol. It is insoluble in water.

Analysis of binary mixtures of normal aliphatic dibasic acids and esters. Use of composition-melting-point relations of the acids. D. F. Houston and W. A. Van Sandt. Indus. and Engin. Chem., Analyt. Ed. 13(9):538-540. Sept., 1946. An empirical method is presented for determining the melting ranges of fused and quenched samples of dibasic acid mixtures. Application to binary mixtures of alternate and adjacent acids containing six to twelve carbon atoms reveals that compositions may be determined within 1 to 5 percent, depending on the composition of the sample. Approximate data on eutectic temperatures of the systems are included.

Analysis of binary mixtures of normal aliphatic dibasic acids and esters. D. F. Houston and J. S. Furlow. Indus. and Engin. Chem., Analyt. Ed. 12(9):541-542. Sept., 1946. Measurements on purified dimethyl esters of dibasic acids having from six to twelve carbon atoms show that their refractive indices bear linear relations to change in temperature and to weight percentage of the composition of binary mixtures. A change of 0.0001 index unit corresponds to three to six percent change in composition of the mixtures, thus approaching by a simple and rapid measurement the accuracy obtainable by saponification equivalent determination.

#### Patent

Process for vapor-phase dehydrochlorination. No. 2,430,897. G. R. Van Atta and D. F. Houston. Patented Nov. 18, 1947. Involves conversion of saturated fatty acids into unsaturated fatty acids, the latter being useful in preparing drying oils. Saturated fatty acid is chlorinated, then subjected to dehydrochlorination in the vapor phase whereby unsaturated product is formed.

## Tartrates and Tannin

### Mimeographed circular:

AIC-14 Recovery of tartrates from grape wastes. Aug., 1943; revised Dec., 1946. Reports research on recovery of tartrates from still slops through use of synthetic anion exchangers placed in beds similar to filter beds.

### Journal articles:

\*Tartrates from grape wastes. Use of ion exchangers in acid-carbonate cycle. J. R. Matchett, R. R. Legault, C. C. Nimmo, and G. K. Notter, *Indus. and Engin. Chem.* 36(9):851-857, Sept., 1944. A substantial part of the approximately 15,000,000 pounds of tartaric acid annually required in our domestic economy and hitherto imported is potentially available in wastes of the grape-processing industry. Laboratory-scale experiments indicate commercial feasibility of utilizing synthetic ion-exchange materials for recovery of this and possibly other valuable constituents of the wastes. Results of experiments involving a chloride-tartrate ion-exchange cycle on the anion exchanger bed thus far indicate that in this cycle the exchanger retains its efficiency longer in contact with the complex grape substance; in addition the process would require only about half the plant setup demanded by the procedure outlined here. The cation exchanger would not be required. In either cycle thorough defecation and decolorization of the influent solutions appear certain to prolong effectively the useful life of exchangers.

Tannin extract from Western hemlock bark. E. F. Potter, K. T. Williams, T. L. Swenson, and I. C. Feustel. *Indus. and Engin. Chem.* 36(12):1146-1149, Dec., 1944. An improved process is suggested for the preparation of tannin extract from the bark of floated Western hemlock logs collected at pulping centers in the Pacific Northwest. A horn-angle hydraulic press was used to prepare the moist bark for extraction. Bark pressed in this manner required only 4 hours for leaching, and the material did not pack or channel in the experimental leaching vats; it would probably leach well in continuous countercurrent-type equipment. Solubilization, found necessary to reduce the high content of insolubles in the tannin extracts, was accomplished by action of sodium bisulfite.

Freezing

## Mimeographed circulars:

- AIC-35 Determination of ascorbic acid in fresh, frozen, and dehydrated foods. Dec., 1943.
- AIC-40 Velva Fruit--A new frozen fruit dessert. Rev. March, 1946.
- AIC-46 Selected bibliography on freezing preservation of fruits and vegetables. 1920-1946. Rev. May, 1947
- AIC-53 Home preparation of Velva Fruit -- A new frozen fruit dessert. July, 1944.
- AIC-57 Commercial preparation and freezing preservation of sliced apples. Aug., 1945
- AIC-120 Sanitation of frozen foods. July, 1946.
- AIC-166 Catechol test for frozen fruits. Dec., 1944.

## Bulletin:

Freezing to preserve home-grown foods. H. C. Diehl and K. F. Warner, U. S. Dept. Agr. Circ. No. 709. Aug., 1945. 62 pages. Includes also directions for meats, poultry, eggs, fish, game, butter, vegetables.

## Journal articles:

- \*Are your cartons moisture-proof? W. Rabak. West. Canner and Packer 33 (11): 52-55. Oct., 1941. Reports a series of experiments which indicate that wraps and liners, not package types, prevent weight loss and hence protect color and quality of frozen peas.
- \*Ascorbic acid. Rapid determination in fresh, frozen, or dehydrated fruits and vegetables. H. J. Loeffler and J. D. Ponting. Indus. and Engin. Chem. Analyt. Ed., 14(11):846-849. Nov., 1942. Reports that ascorbic acid can be determined quickly in fruits and vegetables, whether fresh, frozen, or dehydrated, by disintegrating the sample with dilute metaphosphoric acid in a high-speed cutter and measuring decolorizing effect of extracted ascorbic acid on indophenol dye with a photoelectric colorimeter. The ascorbic acid is distributed within the total liquid phase present, which includes water and dissolved solids originally in the sample. By using a large proportion of extractant and knowing approximate amount of liquid in sample, ascorbic acid can be determined from as little as 3 ml. of filtrate after only one extraction. By using a large amount of 1 percent metaphosphoric acid as extractant the buffering step is avoided, since the pH obtained is sufficiently low to prevent losses during blending, yet sufficiently high to prevent fading of the dye reagent
- \*Preserving fruits and vegetables in frozen food lockers. J. A. Berry. West, Canner and Packer 34(4):50-52. 1942. Deals with selection, preparation, and freezing of chief fruits and vegetables suitable for preservation in cold storage lockers.
- \*Frozen, sliced, crushed, and pureed fruits. D. G. Sorber. Canner 94(7): 16-17. 1942:94(8):18. 1942. Part I of this paper comprises a discussion of basic factors involved in preserving in sliced, crushed, and pureed fruit the natural fresh-fruit flavor and color through the freezing

process. Varietal characteristics, maturity at harvest, transportation, storage, and precooling are dealt with; the preparation of fruit, the contaminative effect of certain metals, and fundamentals of packaging are presented. Part II discusses actual freezing operation, including methods of freezing, rate of freezing, storage temperature, and defrosting. The paper concludes with a discussion of the chief uses of frozen fruits prepared in the various forms specified by the title.

\*Frozen fruits available. D. G. Sorber. Ice Cream Field Year Book. 1942. Presents material on availability of frozen fruits and their suitability for use in ice creams and sherbets.

\*Effects of heat sealing on water-vapor permeabilities of coated cellophanes. W. Rabak and G. L. Dehority. Modern Packaging 17(7):161-163. March, 1944. Emphasizes destructive effects of high temperatures upon coatings and their erratic behavior, possibly due to impairment. Efficiencies of these cellophanes were not impaired by one-second contact between sealing jaws at 385°F. This indicates that the lowest sealing temperature commensurate with firm bonds is desirable. Because coatings vary in behavior during sealing, it is desirable to adjust temperatures to coating types. A sealing temperature of 450°F. was markedly destructive to the three cellophanes, in one case increasing permeability of the heat-modified area 60 times over that of an equivalent area of the untreated sheet.

\*"Fondant-like" formation on fruits caused by crystallization of sucrose. W. Rabak and H. C. Diehl. West. Canner and Packer 36(4):55. April, 1944. Conclusions were that fondant-like formation so frequently encountered in commercially stored frozen "sugar-pack" fruits is due primarily to crystallization of sucrose from the pack. The peculiar "mold-growth" appearance is in all probability due to the slow crystallization of sucrose influenced by conditions not now clearly understood.

\*An analysis of the frozen fruit industry in Utah. D. G. Sorber. Farm and Home Science 5(2):1. June, 1944. Presents economic background of the frozen-food industry, some significant trends to date of writing (1944), and an analysis of the industry in Utah. Fruits treated are apples, apricots, sweet cherries, pears, plums, prunes, blackberries, gooseberries, raspberries, and strawberries. Prices paid for these fruits are briefly discussed in terms of average prices (1927-1942) and medium prices.

Catechol test for frozen fruits. J. D. Ponting. Quick Frozen Foods 7(5): 31. Dec., 1944. Darkening of fruits has long been a source of annoyance to many frozen fruit packers. Fruits that discolor readily, such as apples, apricots, and peaches require pretreatment with a scald, sulfur dioxide or other treatment before they are frozen as a means of inactivating enzymes that cause discoloration. Reduction of enzyme activity controls darkening of fruit flesh before freezing, during frozen storage, and after the fruit is defrosted. The catechol test is generally applicable in determining effectiveness of scalding (with steam or hot water) since in this case the enzyme usually must be completely inactivated before fruit is frozen to prevent browning when it is defrosted. The test is useful in comparing effectiveness of various treatments for inactivation of oxidizing enzymes in fruits.



\*Trends in freezing preservation of foods. V. D. Greaves and M. M. Boggs, Jour. Home Econ. 37(1):23-26. Jan., 1945. (With Univ. Calif) Analyzes trends that were manifest shortly before close of the war and treats of blanching, thawing, nutritive values, and new products including pre-cooked foods.

\*The protective packaging of frozen foods. W. Rabak. Refrig. Engin. 48(5): Nov., 1944: Good Packaging 6(2):21. Feb., 1945. A discussion of factors influencing moisture permeability, packaging materials and package types, packaging and quality retention, specialized packages, and a new method of packaging frozen foods.

\*Impact--effect on moisture barriers at low temperature. W. Rabak and J. B. Stark. Modern Packaging 18(8):137-139. April, 1945. Commercial paraffin-base coatings were more nearly impervious to moisture vapor before subjecting to impact tests than any of four commonly employed heat-sealing over-wrapping materials. Standardized impact test at 70°F. caused greater impairment of sheet-material overwraps than of the dip-coatings. Efficiencies of both the overwraps and the commercial dip coatings were seriously impaired by impact tests at 0°F.

Propionates control microbial growth in fruits, vegetables. E. R. Wolford and A. A. Anderson, Food Indus. 17(6):622-624. June, 1945. Interest in chemical compounds that check microbial growth has led to use of several preservatives in food products and also to research on their mode of action and their effect on foods. Data are presented to show that propionate treatment of figs and berries appreciably retards development of mold. Treatment of shelled peas and lima beans maintains quality longer during delay between harvest and processing. Effect varies with pH.

Determination of sulfur dioxide in fruits. J. D. Ponting and G. Johnson. Indus. and Engin. Chem., Analyt. Ed. 17(11):682-686. Nov., 1945. Sulfur dioxide content of frozen fruits and other types of fruit can be determined rapidly by extraction, blending in buffered sodium chloride solution (which stabilizes sulfur dioxide against enzymic and autoxidation), and acidification and titration with iodine, with and without added formaldehyde, which binds sulfur dioxide.

Retention of ascorbic acid in strawberries during processing, frozen storage, and manufacture of Velva Fruit. H. J. Loeffler. Food Res. 11(1):69-83. Jan., Feb., 1946. Presents data of use in manufacture of Velva Fruit. Effects of condition of fruit, of freezing, of freezing and short storage, of extended storage, and of defrosting were studied in relation to retention of ascorbic acid in sugared and unsugared puree.

Sealing temperature and WVP--A correlation in effectiveness of waxed papers. W. Rabak and J. B. Stark. Modern Packaging 19(8):157-160. April 1946. The heat-sealing operation definitely impairs water-vapor resistance of waxed papers. Extent of impairment increases with elevation of temperature. A sealing temperature of 200°F. is less destructive to water vapor resistance than sealing temperatures of 325 and 450°F. The

latter temperature is excessively destructive. Tests show that machine sealed overwraps of waxed paper are not as efficient in barrier effect as controlled hand-sealed overwraps, which indicates necessity for improvement of mechanical heat-sealing mechanisms. The starch-iodide impregnation method visually demonstrated the effect of varying sealing temperatures on porosity of paraffin-coated papers.

Practical experiments demonstrate protective value of double wrap. W. Rabak and J. B. Stark, West. Canner and Packer 38(11):74-75. Oct., 1946; Food Indus. 18(11):1680-1691. Nov., 1946. Experiments demonstrate that the double over-wrap increases barrier effect. This is particularly obvious in the instance of waxed paper, which resulted in five-fold increase in water-vapor resistance. Efficiency of Cellophane overwrap was increased three-fold. Although this increase may be due in part to offset points of areas of leakage, it is probable that transference of coating from one sheet to another in fluid state during heat sealing is also involved.

‡ How jellied cranberry sauce is preserved by freezing. M. M. Boggs and G. Johnson. Food Indus. 19(8):1067-1069. Aug., 1947. A method of preparing jellied cranberry sauce, without extensive heating, and preserving it by freezing temperature. Freezing weakened gel structure and accentuated syneresis of the gels if prepared without added pectin. Frozen gels, containing 0.25 to 0.35 percent rapid-set, high grade, citrus pectin, were entirely satisfactory in strength and syneresis. The fact that only 50 percent of the added pectin was precipitated under the sugar and pH conditions probably accounted for the decreased syneresis. All rapid-set, citrus pectins tried were satisfactory. Results were not uniform when rapid-set, apple pectin, or slow-set apple and citrus pectins were used.

‡ New fresh-fruit spreads preserved by freezing. G. Johnson and M. M. Boggs. Food Indus. 9(11):1491-1494. Nov., 1947. Two new gelled fruit products, which retain to a high degree fresh flavor and aroma and natural color of ripe fruit, are described. The high retention of fresh flavor is accomplished by preparing the product at room temperature and preserving it at 0°F. or lower. The new products are prepared with fresh or frozen fruit juice or puree, sugar, and pectin. They have a soluble solids content lower than that of jellies and jams.

## FRUITS

### Dehydration

#### Bulletins:

Preservation of fruits and vegetables by commercial dehydration. U. S. Dept. Agr. Circ. 619. 46 pages. Feb., 1942.

Commercial dehydration of vegetables and fruits in wartime. U. S. Dept. Agr. Misc. Pub. 524. 29 pages. Sept., 1943

Vegetable and fruit dehydration. A manual for plant operators. U. S. Dept. Agr. Misc. Pub. 540. 218 pages. June, 1944. (Out of print.)

Journal Articles:

\*Ascorbic acid. Rapid determination in fresh, frozen, or dehydrated fruits and vegetables. H. J. Loeffler and J. D. Ponting. (See under Freezing).

\*Extraction of ascorbic acid in plant materials. Relative suitability of various acids. J. D. Ponting. Indus. and Engin. Chem., Analyt. Ed. 15(6): 389-391. June, 1943. The stabilization of ascorbic acid during its extraction and determination has long been a problem. Of the 13 acids compared as to stabilizing effect on ascorbic acid solutions under conditions favorable to oxidation, only metaphosphoric and oxalic acids appeared suitable, these two acids being far superior to any of the others and about equally satisfactory. It is concluded that oxalic acid may be safely substituted for metaphosphoric acid in the determination of ascorbic acid, thus providing a more stable, more easily obtainable, and less expensive extractant.

Determination of sulfur dioxide in dehydrated foods. A. N. Prater, C. M. Johnson, M. F. Pool, and G. Mackinney. Indus. and Engin. Chem., Analyt. Ed. 16(3):153-157. March, 1944. (With Univ. Calif.) As a control measure in the application of sulfite solutions to vegetables for dehydration, a simple method for determining the sulfur dioxide content has become necessary. The method proposed in this paper was designed for inspection and field work with a minimum of equipment, but it is also well adapted to laboratory research. It has been checked against other methods and also by the polarograph.

The relation of the sulfur dioxide and total sulfur contents of dried apricots to color change during storage. D. G. Sorber. Fruit Prod. Jour. and Amer. Food Mfr. 23(8):234-237, 251. Apr., 1944. This paper presents the results of storage studies conducted at the Laboratory of Fruit and Vegetable Chemistry in Los Angeles. Four sets of apricots, each set representing three lots, were dried during the 1930 season for use in storage tests. The sulfuring process applied to fruit of the different sets varied as to concentration of sulfur dioxide in the atmosphere to which the fresh apricot halves were exposed and as to the duration of exposure. Sulfur dioxide, sulfate sulfur, and total sulfur were quantitatively determined in all samples after 5 days of drying, and after 17 months and 10½ years of storage. Unsulfured fruit was dull and dark brown. Sulfured apricots after prolonged storage were black on the surface and had a pronounced odor and slight flavor of caramelized sugar. The freshly torn surfaces of the sulfured fruit pieces were dark brown.

\*Progress in spray drying of fruits and vegetables. J. R. Matchett. West. Canner and Packer 38(1):71-72. Jan., 1946. Spray drying is a process designed to desiccate almost instantaneously products which would be harmed by prolonged exposure to high temperatures or those whose characteristics do not lend themselves well to other means of dehydration. The process of spray drying, which is used extensively for milk and eggs, may prove to be well adapted to the removal of moisture from other fluid products that are easily injured by heat. In this process dehydration is accomplished within a few seconds by suspending finely atomized fluid in a stream of

hot air and collecting the resulting dry product as a powder.

Dehydrofreezing--new way of preserving food. L. B. Howard, and H. Campbell, Food Indus. 18(5):674-676. May, 1946. Experiments with several fruits and vegetables have shown that moderate dehydration (removal of about two-thirds of the water content) followed by freezing resulted in excellent products in most cases. Advantages of this new method are savings in weight, space, packaging materials, and refrigeration load. The preliminary experiments dealt with potatoes, peas, carrots, asparagus, cherries, boysenberries, and apricots.

## POULTRY BYPRODUCTS

### Feathers

Separation and determination of protein sulfur, sulfide sulfur, and other sulfur in sodium sulfide dispersions of keratins. E. F. Potter and C. B. Jones. *Indus. and Eng. Chem., Analyt. Ed.* 15(1):15-17. Jan., 1943. Sulfur in sodium sulfide dispersions of keratins can be separated into three parts: protein sulfur, sulfide sulfur, and other sulfur compounds by means of basic aluminum acetate. Sulfides can be separated from sulfites, thiosulfates, and sulfates, but not from bisulfites, by treatment with basic aluminum acetate.

\*New plasticizers for vinyl resins and cellulose esters. D. W. Elam, H. M. Preusser, and R. L. Page. *Modern Plastics* 20(9):95-97. May, 1943. Among the plasticizers reported, two succeeded in plasticizing cellulose acetate. Xylitol penta-acetate shows excellent retention, but is unstable when exposed to water. It is a nonsolvent for cellulose acetate. Diethoxyethyl fumarate is retained below 40 percent, is fairly stable and relatively insoluble in water and is a solvent for cellulose acetate at molding temperatures. Both of these plasticizers impart good flow under molding conditions. In cellulose acetate butyrate, xylitol pentabutyrate has the desirable qualities of imparting good flow and good flexibility to the molded piece, is stable and water insoluble and is retained to a high degree. Plasticized pieces are odorless if the plasticizer content is kept below about 50 percent; at 80 percent a slight "butyric" odor can be detected. Dibutoxyethyl fumarate shows excellent stability, is water insoluble, and imparts good flow at molding temperatures.

The dispersion of keratins. I. Studies on the dispersion and degradation of certain keratins by sodium sulfide. C. B. Jones and D. K. Mecham. *Arch. Biochem.* 2(2):209-223. June, 1943. The effects of temperature, time, sodium sulfide concentration, and ratio of keratin to sodium sulfide upon extent of dispersion of feather keratin in solution and upon yield of material obtained by acidification of such dispersions are reported. The following combination of factors was found to yield maximal dispersion with minimal degradation of the protein: digestion with 0.1M sodium sulfide (100 ml. of solution per 7.5 gm of keratin) for about 2 hours at 30°C. The dispersed protein was recovered nearly quantitatively by acidification of the dispersion to pH 4.2. Feather keratin is more readily dispersed and less stable in solutions of sodium sulfide than hoof, hog hair, and wool.

The dispersion of keratins. II. Studies on the dispersion of keratins by reduction in neutral solutions of protein denaturants. C. B. Jones and D. K. Mecham. *Arch. Biochem.* 3(2):193-202. Dec., 1943. In this study it was possible to suppress protein degradation to such an extent that the dispersed proteins could be recovered nearly quantitatively by acid precipitation. Keratins can be dispersed at neutral reaction by cleavage of their disulfide bonds, either by reduction with sulfhydryl compounds or by action of bisulfite, in the presence of any of a number of substances (urea, guanidine, a synthetic detergent, and others) which act upon non-keratin proteins as denaturants. The dispersibility of one keratin may differ from that of another in a particular combination of dispersing and

disulfide-splitting agents and may vary with different combinations of these agents. Of keratins investigated, feather keratin was most readily dispersed in neutral solutions, and ovokeratin was unique in its resistance to dispersion not only in neutral but also in alkaline reducing solutions.

Cystine content and enzyme digestibility of powdered hoof proteins. H. S. Olcott. Proc. Soc. Expt. Biol. and Med. 54 (2):219-220. 1943. Considerably more than half of dried powdered cattle, horse, and dog hoof material was found to be digestible and horse hoof the least. The undigestible residue in each case contained more cystine than the original preparations. The soft part of cattle hoof contained less cystine and was more readily digested than the horn-like front portion. Thus hoof powder consists of at least two and possibly a number of different kinds of proteins.

Artificial fibers from corpuscular and fibrous proteins. H. P. Lundgren and R. A. O'Connell. Indus. and Engin. Chem. 36(4):370-374. April, 1944. Proteins in general are potential fiber-building material. By proper manipulation of proteins from surplus and waste commodities fibers have been made with molecular orientation, dry strength, and moisture-absorbing characteristics comparable with those of natural protein fibers.

Esterification of fatty and amino acids with 1,2-epoxides in aqueous solution. H. Fraenkel-Conrat and H. S. Olcott. Jour. Amer. Chem. Soc. 66 (8):1420. Aug., 1944. Model experiments were performed in which fatty acids and amino acids were treated with ethylene oxide, 1,2-propylene oxide, or epichlorohydrin in aqueous solution or suspension at room temperature. In contrast to carboxyl groups, amino groups appeared to react more readily in the uncharged state, i.e., in alkaline solution, or only after all acids originally present had been "neutralized" by combination with the epoxide. Complete disappearance of primary amino groups in 0.08 molar solutions of monosodium glutamate or of alanine in presence of sodium acetate (0.08 molar) occurred within two days of treatment with excess propylene oxide (3 molar). The dipolar ion did not react as readily in absence as in presence of other electrolytes.

\*Keratin--A modifier for phenolic plastics. G. H. Brother, C. H. Binkley, and B. Brandon. Modern Plastics 22(7):157-160, 196, 198. March, 1945. Describes properties and advantages of keratin as a modifier for phenolic plastics. Readily available keratins such as feathers, hoofs, and hair can be used, after simple treatment, to modify phenolic molding powders. The modified powder can be prepared by the same method and the same equipment as used for preparation of unmodified phenolic-resin molding powders. It has practically the same flow properties as general-purpose phenolics; hence it is readily adapted to standard molding procedure and equipment, but may require a somewhat longer molding cycle to insure cure. The finished molded product absorbs somewhat more water than do the molded products of most phenolics. Its flexural strength is about 15 percent less than that of unmodified phenolic plastic, and its impact strength is about the same. The phenolic-keratin plastic can be produced in a wider range of colors than phenolics, and its arc resistance is at least two or three times as great.

Synthetic protein fibers from protein-detergent complexes. H. P. Lundgren. Textile Res. Jour. 15(10):335-353. Oct., 1945. Use of hydrocarbon salts in preparation of spinning dispersions from corpuscular as well as fibrous proteins is described. Egg-white and chicken-feather keratin were used as typical proteins. By appropriate stretching along with removal of detergent, highly oriented fibers having the beta-keratin structure typical of natural protein fibers have been made. With crystalline egg albumin as a model protein, it is shown that primary interaction between proteins and long-chain hydrocarbon salts is electrostatic, involving basic groups of protein when anionic detergents are used. Additional detergent can be bound, presumably by residues of the detergent bound electrostatically and by nonpolar forces. When additional detergent is bound, the complex acquires flow properties necessary in the fiber-making process.

Physicochemical characterization of dispersed chicken feather keratin. W. H. Ward, L. M. High, and H. P. Lundgren. Jour. Polymer Res. 1(1):22-36. Jan., 1946. Chicken feather keratin, dispersed in a neutral aqueous solution of an alkyl-benzenesulfonate with sodium bisulfite, forms with sulfonates a stable complex, which was freed from dialyzable material. The dialyzed complex contained about three parts of protein to two of sulfonate. The specific refractive increment of the complex was  $199 \times 10^{-5}$  per gm. per 100 cc. ( $= 539$  mu); a representative value for the partial specific volume of the complex is 0.801 cc per gm. in dilute aqueous solution at 28.8°C. Electrophoresis shows two principal electrophoretically distinct species, in approximately equal quantity, having falling mobilities less than that of the free sulfonate. A representative number-average molecular weight, determined from osmotic pressure, is 57,000. The normalized diffusion gradient is symmetrical but is leptokurtic, deviating considerably from the ideal. The weight-average diffusion constant is about  $6.91 \times 10^{-7}$  cm.<sup>2</sup> per sec., referred to pure water at 20°C. Gralen's ratio of the diffusion constants was found to be 1.35. The material is heterodisperse. On sedimentation analysis, the complex exhibited a single boundary having considerable spread. The average sedimentation constant, extrapolated to infinite dilution and referred to pure water at 20°C., is 5.18 Svedberg units. The average molecular weight determined from the sedimentation constant and the weight-average diffusion constant is 75,100. The computed frictional ratio,  $f/f_0 = 1.07$ , of the complex shows that it is not extended in dilute solution. If the representative molecular unit is an unhydrated prolate ellipsoid of revolution, the ratio of the major to minor axis is 4 and the absolute values of the length and width of the unit are 106 and 42.4 Å. The molecular weight of the keratin portion of the complex is of the order of 34,000 to 40,000, depending upon the average chosen.

### Patents

Process for producing glues and adhesives from keratin protein materials. Patent 2,399,161 to G. H. Brothier and C. H. Binkley, Patented April 30, 1946. Adhesive made by reducing a keratin material with an alkaline sulphide solution, dispersing the product in caustic soda and drying it. Process enables waste keratin materials such as feathers, animal hair, etc. to be converted into useful products.

†Process of making oriented regenerated protein products. Patent 2,425,550 to H. P. Lundgren. Patented Aug. 12, 1947. Process of preparing films or fibers from protein materials. Waste keratin materials such as feathers, animal hair, etc. may be used as the raw material. The process essentially involves dispersing the protein in a detergent solution, precipitating a protein-detergent complex, forming a film or fiber therefrom by extrusion and then extracting the film or fiber with solvent to remove the detergent.

### Egg White

The formation of fibers from nonfibrous native proteins. H. P. Lundgren. Jour. Amer. Chem. Soc. 63(10):2854-2855. October, 1941. This note concerns use of detergents by means of which it has been found possible to control transformation of native forms into unfolded configuration of all nonfibrous proteins so far tested in this Laboratory. This change was followed in two ways: by the ability to draw fibers from the precipitated protein and by corresponding viscosity changes in the system protein-detergent. Elastic and highly double-refracting fibers were obtained in this manner from the following proteins: crystalline egg albumin, purified preparations of hog thyroglobulin, wheat glutenin, casein, and commercial preparations of zein and blood albumin. Detergents appear to be superior to other reagents which have been investigated, although some, including pyrogalllic acid and guanidine hydrochloride, were found to behave somewhat similarly. The conditions favorable for drawing of fibers appear to vary with the protein, nature and concentration of reagents, temperature and pH of the system.

Electrophoretic study of the action of alkylbenzene sulfonate detergents on egg albumin. H. P. Lundgren, D. W. Elam, and R. A. O'Connell. Jour. Biol. Chem. 149(1):183-193. July, 1943. Conclusions from electrophoretic analysis of an egg albumin-alkylbenzene sulfonate system: Both native and heat-denatured egg albumins form complexes of well-defined electrophoretic patterns with alkylsulfonates in solutions alkaline to the protein isoelectric point. In mixtures containing large amounts of native protein, detergent combines with a maximum of 3 times its weight of protein, leaving excess native albumin free. When heat-denatured protein is mixed in large amounts with detergent, there is complete combination in all ratios. Complexes formed in solutions containing a large amount of detergent have a correspondingly high detergent content; however, some of this detergent is reversibly bound, since dissociation of detergent from complex occurs during electrophoresis. This phenomenon occurs in complexes formed by mixing with either native or denatured albumin. The protein-detergent complex exhibits an electrophoretic mobility, within limits, that is parallel to the composition of the complex. Protein in the complex exhibits the electrophoretic behavior of completely denatured protein when the fraction of detergent present is 0.3 or more.

The molecular structure of fibers made from native egg albumin. K. J. Palmer and J. A. Calvin. Jour. Amer. Chem. Soc. 65(11):2187-2190. Nov., 1943. Results of an X-ray diffraction study of these fibers are reported. Fibers made from native egg albumin by a process which involves complex formation



with detergent and drawing under steam are shown to be composed of parallel bundles of polypeptide chains running parallel to the fiber axis. The peptide chains have the beta-keratin configuration, as is evident from the similar appearance of X-ray patterns obtained from the albumin fibers as compared with those obtained from well-oriented beta-keratin. The tensile strength of synthetic pure protein fiber is shown to be dependent upon degree of molecular orientation and reaches a value of 38,000 pounds per square inch.

The structure of an egg albumin-detergent complex. K. J. Palmer. Jour. Phys. Chem. 48(1):12-21. Jan., 1944. From the structure postulated in this study the author suggests that the polar amino acids probably alternate along the peptide chain in egg albumin. On the basis of this hypothesis it follows that if the layer structure for native egg albumin postulated by Pauling is acceptable, the layers in the native configuration are polar. By use of this model a plausible structure is postulated for the complex between native egg albumin and detergent. This structure appears to account for the observed facts that the complex formed from native egg albumin never contains less than 25 percent of Nacconol NRSF (whereas that formed from denatured egg albumin does), and that the complex containing 75 percent native egg albumin and 25 percent detergent can exist in equilibrium with free native protein.

Artificial fibers from corpuscular and fibrous proteins. (See under Feathers.)

Synthetic protein fibers from protein-detergent complexes. (See under Feathers.)

Water resistance of proteins. Improvement through chemical modification. H. S. Olcott and H. Fraenkel-Conrat. Indus. and Engin. Chem. 38(1):104, 106. Jan., 1946. Proteins were treated with a number of organic reagents and the products examined for water resistance by measurement of the uptake of water by pressed disks. Aryl and long-chain alkyl isocyanates and also aromatic acid anhydrides and chlorides proved most effective. A number of proteins yielded phenyl isocyanate derivatives showing 24-hour water absorption of 1 to 2 percent. Phthalic anhydride gave products of low water absorption with egg white and cattle hoof. Protein derivatives of low water absorption showed a tendency to plastic flow without the addition of water as plasticizer.



## VEGETABLES

### Freezing

#### Mimeographed circulars:

- AIC-10 Frozen pork and beans of the tomato sauce type. June, 1943.  
AIC-34 A test for adequacy of blanching in frozen vegetables.  
Revised May, 1947.  
AIC-35 Determination of ascorbic acid in fresh, frozen, and dehydrated foods. Dec., 1943.  
AIC-36 Freezing preservation of pumpkin pie stock. Dec., 1943.  
AIC-46 Selected bibliography on freezing preservation of fruits and vegetables, 1920-45. Rev. No. 3. May, 1947.  
AIC-120 Sanitation of frozen foods. June, 1946.

#### Bulletin:

Freezing to preserve home-grown foods. H. C. Diehl and K. F. Warner. U.S.D.A. Circ. 709. 62 pages. 1945. Directions for preparation, storage, and handling of meats, poultry, eggs, fish, game, butter, vegetables, and fruits, particularly for locker patrons and users of home freezing cabinets.

#### Journal articles:

Notes on the tenderometer. H. Campbell. West. Canner and Packer 31(6):113-114. May, 1939. Rapid growth of the frozen pea industry shows the need for quality control and standardization. Discusses usefulness of tenderometer in grading peas.

The splitting of shelled peas intended for freezing. H. Campbell. West Canner and Packer 32(8):49-50. July, 1940. Presence of split and otherwise damaged peas materially detracts from appearance of frozen product, and, if severe, may constitute a grade defect. Data show that initial cause for excessive splitting of shelled peas seems to lie in improper viner operation. Since increase in splitting takes place on holding of peas in iced or uniced condition, it would seem that avoidance of delays would tend to reduce damaged peas in pack. Although scalding techniques may account for some splitting, their significance seems to be relatively unimportant; the advantage seems to be with steam scalding and, where water scalding is employed, at temperatures below or at 200°F.

\*Scalding of cut corn for freezing. H. Campbell. West. Canner and Packer 32(9):51-53. Aug., 1940. Primary purpose underlying scalding of corn is, of course, to inactivate enzymes which otherwise would bring about off flavors, odors, and colors during storage. Because little seemed to be known relative to minimum scalding requirements for cut corn, experiments were undertaken to determine this point.

\*Quality in frozen pack peas. H. Campbell and H. C. Diehl. West. Canner and Packer 32(10):48-50. Sept., 1940:32(11):51-53. Oct., 1940. Factors that influence quality, maturity, handling, variations within pack, and standards for grades are discussed.

\*Technological aspects of locker plant industries. H. C. Diehl. Quick Frozen Foods: I. 3(7):16-17. 1941: II. 3(8):24, 37-38. 1941. Emphasizes importance of locker plant in food conservation, increasing use, relationship to frozen foods industry, necessity for uniform 0°F. of slightly lower temperature, significance of sharp freezing facilities and plant improvement, desirability of dispensing information to patrons regarding preparation of raw materials and use of frozen materials, value of uniform practice in locker industry, and need for a uniform, clearly objectified policy for the industry as a whole.

\*Are your cartons moisture-proof? W. Rabak. West. Canner and Packer 33(11):52-55. Oct., 1941. Report of a series of experiments which indicate that wraps and liners, not package types, prevent weight loss and hence protect the color and quality of frozen peas.

\*The fewer the bacteria, the better the frozen pack. J. A. Berry. Canner 94(4):13-14. 1941. Describes food spoilage as produced by bacteria and other micro-organisms, sources of contamination, significance of bacterial counts, and kindred topics.

\*Temperature and tenderometer. How temperature may affect tenderometer value for peas. H. Campbell. West. Canner and Packer 34(2):39-40. Feb., 1942. While work reported was preliminary, it seems apparent that temperature of peas may affect tenderometer values and is a factor which should be considered. Temperature effect points to desirability of standardized procedure wherein samples would be brought to a uniform temperature by tempering in water. Degree of hardness of water need not be considered.

\*Can frozen foods help win the war and write the peace? H. C. Diehl. West. Frozen Foods 3(4):5-6,8,10. 1942. An analysis of part that frozen foods might play in war. Strategic advantages of frozen foods, particularly saving of packaging materials such as tin.

\*Preserving fruits and vegetables in frozen food lockers. J. A. Berry. West. Canner and Packer 34(4):50-52. 1942. Selection, preparation, and freezing of chief fruits and vegetables suitable for preservation in lockers.

\*Packaging of frozen foods under war conditions. H. C. Mehl and W. Rabak. Proc. Inst. Food Technol., pp. 117-120. 1942. Efficient packaging to guard against loss of moisture and oxidative changes in frozen foods and thus insure quality retention is discussed. Glazing is suggested as one important means of retaining quality.

Factors influencing the texture of peas preserved by freezing. M. M. Boggs, H. Campbell, and C. D. Schwartze. Food Res. \*I. 7(4):272-287. July-Aug., 1942. II. 8(6):502-515. Nov.-Dec., 1943. (With Wash. Agr. Expt. Sta.) Part I: The specific texture investigations reported are: (1) variation for a given sieve size and variety of peas grown in the same field and harvested on the same day; (2) variation for a given

sieve size and variety of peas grown in the same field but harvested on different days of the same season; (3) effect of cooking for 5, 10, and 15 minutes; (4) effect of vining; (5) effect of delay between vining and freezing.

Part II: Varieties suitable for freezing were grown under commercial conditions, vined, cleaned, scalded for one minute in water at 210°F., cooled in water to approximately 57.2°F., packaged in 12-ounce waxed-paper cartons, wrapped, and heat-sealed in moisture-vapor-proof Cellophane, then frozen and stored at 0°F. After several months the peas were cooked without preliminary thawing in boiling water for six minutes, and the texture of the skins of all samples was determined by measuring load in kilograms required to penetrate three layers of skins with a one-eighth-inch, steel, ball-bearing penetration point. In one study texture of cotyledon was also determined by measuring load required to crush one cotyledon to one-fourth of its thickness.

\*Frozen foods have good health record. J. A. Berry. Quick Frozen Foods 6(3):46. 1943. A semitechnical article, pointing out that frozen foods have not been nor are likely to be responsible for any outbreak of food poisoning.

\*Delay affects frozen pea quality. J. P. Nielsen, E. R. Wolford, and H. Campbell. West. Canner and Packer 35(6):47-48. June, 1943. This evaluation of losses in quality constituents in shelled peas delayed between harvest and freezing shows that, when peas are held at an average temperature of 76°F., serious losses do not occur in 4 hours, but that beginning at 8 hours there is rapid deterioration in sugar, vitamin C., flavor, and texture, accompanied by great increases in bacterial content. Peas delayed 12 hours at 76°F. are unfit to pack, and even an 8-hour delay is to be avoided. Use of ice or any other method of reducing temperature is strongly recommended during such delays and may be routinely necessary in many instances. A reduction in temperature of raw product, if only to 50°F., as in the work described, is of considerable benefit, especially in checking growth of bacteria which with natural respiration of the product are, as is well known, responsible for loss of sugar and lack of fresh flavor.

\*Freezing baked beans and other prepared foods. D. G. Sorber. Quick Frozen Foods 5(8):18-19, 24. 1943. Report of work done on freezing baked beans. Aspects of preparation, evaluations of color and flavor, nutritional value, and storage behavior are discussed.

\*Direct microscopic method to estimate sanitary history of frozen pack peas, E. R. Wolford. West. Canner and Packer 35(13):58. Dec., 1943. This paper enumerates advantages of direct test over plate-count or cultural method for frozen food products.

\*Effects of heat sealing on water-vapor permeabilities of coated cellophanes. W. Rabak and G. L. Dehority. Modern Packaging 17(7):161-163, 220. Mar., 1944. Emphasizes destructive effects of high temperatures upon coatings and their erratic behavior, possibly due to impairment. The efficiencies of these cellophanes were not impaired by one-second contact between sealing jaws at 285°F. However, permeability of the heat-

modified areas was increased 3 to 8 times when sealing surfaces were maintained at a temperature of 385°F. Because coatings vary in behavior during sealing, it is desirable to adjust temperatures to coating types. A sealing temperature of 450°F. was markedly destructive to the three cellophanes, in one case increasing permeability of the heat-modified area 60 times over that of an equivalent area of untreated sheet.

\*Frozen tomatoes not too good, juice has possibilities. Food Indus. 16(8):532-533. Aug., 1944. Frozen juice has good color and pleasant flavor, but tomato sections collapse on thawing. General intention is to suggest means of improving processing frozen tomatoes and juice.

\*The protective packaging of frozen foods. W. Rabak. Refrig. Engin. 48(5): Nov., 1944; Good Packaging 6(2):21. Feb., 1945. Discussion of factors influencing moisture permeability, material and types, quality retention, specialized packages, and a new method of packaging frozen foods.

\*Impact--effect on moisture barriers at low temperature. W. Rabak and J. B. Stark. Modern Packaging 18(8):137-139,166. April, 1945. Commercial paraffin-base coatings were more nearly impervious to moisture vapor before subjection to impact tests than any of four commonly employed heat-sealing over-wrapping materials. Standardized impact test at 70°F. caused greater impairment of the sheet-material overwraps than of the dip-coatings. The efficiencies of both the overwraps and the commercial dip-coatings (except coating D) were seriously impaired by impact tests at 0°F.

Propionates control microbial growth in fruits and vegetables. E. R. Wolford and A. A. Andersen. Food Indus. 17(6):622-624. June, 1945. Interest in chemical compounds that check microbial growth has led to use of several preservatives in food products and also to research on mode of action and effects on foods. Presents data to show that propionate treatment of figs and berries appreciably retards development of mold. Treatment of shelled peas and lima beans maintains quality longer during delay between harvesting and processing. Effect varies with pH.

Sealing temperature and WVP--A correlation in effectiveness of waxed papers. W. Rabak and J. B. Stark. Modern Packaging 19(8):157-160. April, 1946. Heat sealing definitely impairs water-vapor resistance of waxed papers. Extent increases with elevation of sealing temperature. A temperature of 200°F. is less destructive to water-vapor resistance than 325 and 450°F. The latter is excessively destructive. Tests showing that machine-sealed overwraps are not as efficient as controlled hand-sealed overwraps indicate necessity for improvement of mechanical heat-sealing mechanisms. The starch-iodide impregnation method visually demonstrates effect of varying sealing temperatures on porosity of paraffin-coated papers.

Practical experiments demonstrate protective value of double wrap. W. Rabak and J. B. Stark. West. Canner and Packer 38(11):74-75. Oct., 1946; Food Indus. 18(11):1680-1691. Nov., 1946. Double overwrap increases barrier effect, particularly obvious in waxed paper, which was increased five-fold in water-vapor resistance. Efficiency of cellophane overwrap

was increased three-fold. Although this increase may be due in part to off-set points or areas of leakage, it is probable that transference of coating from one sheet to another in the fluid state during heat-sealing is also involved. Known facts regarding destruction of coatings during heat-sealing imply that second over-wrapping operation results in partial resealing of the porous areas of first overwrap. The extensive areas of impaired coating shown by the starch-iodide method suggests latter possibility.

Ontogeny of the sclereids in the integument of Pisum sativum L. R. M. Reeve. Amer. Jour. Bot. 33(10):806-816. Dec., 1946. Detailed histological account of development of sclereids of peas is given. Structural changes involve changes in composition of cell wall materials and middle lamellae. Maturity stages ranging from extremely immature, through succulent stages used in freezing are included in these studies.

†Relation of histological characteristics to texture in seed coats of peas. R. M. Reeve. Food Res. 12(1):10-23. Jan., 1947. Major causes of texture change during maturation is formation of specialized cell walls in epidermal cells (sclereids). Other structures and their significance to texture and maturity are discussed. Brief experiments on mineral nutrition showed that (without change in skin thickness) plants grown in high Ca (low K) produced peas with skins tougher than those from control plants, and plants grown low Ca (high K) produced peas with skins more tender than those of control. Definition and distinction between inherent and environmental factors of texture is made on a histological basis.

†Degree of maturity influences the quality of frozen peas. J. P. Nielsen, H. Campbell, C. S. Bohart, and M. P. Masure. Food Indus. I. 19(3): 81-84. Mar. 1947; II. 19(4):103-106. Apr. 1947. Part I: Sugar and fat determinations do not appear suitable as indexes of maturity. Protein and skin texture offer possibilities. Starch content shows promise of usefulness as an objective measure of maturity and texture. Part II: Total solids content and starch content are two good objective indexes of the maturity of peas. Limits are suggested for the starch and total solids contents for the fancy, extra standard, and standard grades.

†Effect of flavonols on Clostridium botulinum. A. A. Andersen and J. A. Berry. Science 106(2765):644-645, Dec., 1947. In tests of rutin, quercitrin and quercetin against Clostridium botulinum it was found that quercetin in concentrations of 120 to 160 ppm. prevented growth for 10 days at least. It did not appear to destroy pre-formed botulinum toxin. Rutin had no effect, and quercitrin had but slight inhibitory effect on growth of Cl. botulinum. The effect of quercetin on an anaerobic organism has not previously been reported.

#### Patent

Device for peeling agricultural products. No. 2,403,923. G. T. Hemmeter. Patented July 16, 1946. Device consists essentially of a furnace through which products, potatoes, for example, are conveyed. A mechanism is provided to tumble the products in their passage through furnace whereby

entire surface is exposed to radiant heat to char skin. Charred skin is readily removed by water sprays.

### Constituents and Analytical Methods

Protein and sulfur content of immature lima beans as affected by varietal and environmental factors and processing. M.E. Davis, E. M. Chace, and D. G. Sorbor (With Univ. Calif.). Food Res. 7(1):26-37. 1942. Concerned with protein and sulfur contents of green lima beans as affected by hereditary and environmental factors. There is slight change in the nitrogen and sulfur fractions of the total solids because of removal of white, immature beans or because of blanching, freezing, and drying processes.

Vitamin A value of fresh and dehydrated carrots. R. H. Wilson, J. O. Thomas, and F. DeEds. Food Prod. Jour. 22(1):15-17. Sept., 1942. Concerned with Chantenay variety. The parallelism between chemical and biological methods for evaluating vitamin A both before and after a processing technique was studied.

\*Ascorbic acid. Rapid determination in fresh, frozen, or dehydrated fruits and vegetables. H. J. Loeffler and J. D. Ponting. Indus. and Engin. Chem., Analyt. Ed. 14(11):846-849 1942. Ascorbic acid can be determined quickly in fruits and vegetables, whether fresh, frozen, or dehydrated, by disintegrating sample with dilute metaphosphoric acid in high-speed cutter and measuring decolorizing effect of the extracted ascorbic acid on indophenol dye with a photoelectric colorimeter. The ascorbic acid is distributed within the total liquid phase present, which includes the water and dissolved solids originally in the sample. Hence by using large proportion of extractant and knowing the approximate amount of liquid in the sample, the ascorbic acid can be determined from as little as 3 ml. of filtrate after one extraction. By use of a large amount of 1 percent metaphosphoric acid as extractant the buffering step is avoided, since pH obtained is sufficiently low to prevent losses during blending, yet sufficiently high to prevent fading of the dye reagent. Rapid determinations can be made easily on highly pigmented berries and tough dehydrated vegetables.

\*A new method for the determination of moisture in dehydrated vegetables. B. Makower and S. Myers. Proc. Inst. Food Technol. pp. 156-164. 1943. Experiments are described to show how seriously results are affected by fineness of grinding of sample and by time of heating, temperature, and pressure. Carrots ground to 40 mesh (optimum fineness) require at least 40 hours for accurate determination. It is therefore proposed that vapor pressure rather than percentage of water be adopted as an index of moisture level. A simple apparatus is described for measurement of vapor pressure. Advantages are that sample does not require weighing, results are independent of fineness of grinding, and measurement may be completed in approximately two hours. The relations between vapor pressure and moisture content are shown for dehydrated carrots and spray-dried eggs.

\*Equilibrium moisture content of dehydrated vegetables. B. Makower and G. L. Dehority. Indus. and Engin. Chem. 35(2):193-197. Feb., 1943. The



equilibrium moisture content of some dehydrated vegetables was determined by allowing them to attain equilibrium in air-free desiccators containing sulfuric acid solutions to control relative humidity. Adsorption and desorption measurements were carried out on unblanched carrots, cabbage, sweet potatoes, spinach, and white potatoes. The sorption isotherms for all the vegetables are S-shaped, and are characterized by an inflection point in the neighborhood of 5 percent moisture content. Measurements were also made on blanched white potatoes. Blanching causes a decrease in equilibrium moisture content. The decrease is ascribed to a change in physical state of starch granules in the potato.

\*Rapid determination of starch in vegetables. J. P. Nielsen. Indus. and Eng. Chem., Analyt. Ed. 15(3):176-179. March, 1943. In certain vegetables such as peas, corn, and lima beans, the starch content of the seed tends to increase as the plant matures. This increase is usually associated with a decrease in total sugar and tenderness. A rapid and reasonably accurate method for determination of starch in certain vegetables has been developed. It includes grinding fresh sample, extracting starch with perchloric acid, and estimating by photoelectric colorimeter the dissolved starch indicated by the blue color produced with iodine. Alcohol extraction of products studied was found unnecessary. Use of red filter in colorimeter considerably reduces error produced by dextrans when present.

\*A microscopic study of physical changes in carrots and potatoes during dehydration. R. M. Reeve. Food Res. 8(2):128-136. March-April, 1943. Preparation of vegetable tissues for microscopic examination after different periods and techniques of drying was accomplished by a modified celloidin technique by which dehydrated condition was preserved. Extent of shrinkage during dehydration and differences between blanched and unblanched, dehydrated vegetables are described. Case hardening is conditioned by rapidity of evaporation, cell contents, and thickness of slices or cubes. Blanching of potato causes starch gelation, and gelled starch seals dehydrated tissues.

\*Microscopy of oils and carotene bodies in dehydrated carrots. R. M. Reeve. Food Res. 8(2):137-145. March-April, 1943. Blanching and dehydration break down lipoprotein and free oil in which some carotene dissolves. Off odors of dehydrated carrot are related to oil and carotene oxidation. Preservation of carotene in the blanched, dehydrated carrot is partially effective according to amount of carotene dissolved in and coated by oil, and also according to rate at which oils oxidize. Unblanched, dehydrated carrots contain less oil-protected carotene. Blanching causes earlier and more complete liberation of oil, which coats and protects carotene.

\*Changes in tissue composition in dehydration of certain fleshy root vegetables. R. M. Reeve. Food Res. 8(2):146-155. March-April, 1943. Microscopic evidence correlated with chemical analyses indicates that preservation of carotinoids in certain vegetables is favored by blanching before dehydration. Histological studies show a definite correlation between blanching and rate of carotene oxidation upon storage of dried product, particularly in starchy vegetables. This correlation is established by comparisons of low-starch vegetables with starchy vegetables as to oil protection of carotene and as to protective action of gelled starch on

both oils and carotene in sweet potatoes. Blanching denatures cytoplasm of raw slices and a greater amount of pectin solution is soaked up by blanched slices than by raw.

\*Tenderizing vegetables for freezing. J. P. Nielsen, H. Campbell, and M. Boggs. West. Canner and Packer 35(6):49. June, 1943. Experiments reported in this paper show blanching in solution of sodium hexametaphosphate to be an effective method of tenderizing.

\*Extraction of ascorbic acid from plant materials. Relative suitability of various acids. J. D. Ponting. Indus. and Engin. Chem., Analyt. Ed. 15(6):389-391. June, 1943. Of 13 acids compared as to stabilizing effect on ascorbic acid solutions under conditions favorable to oxidation, only metaphosphoric and oxalic acids appeared suitable, these two being far superior to others and about equally satisfactory. It is concluded that oxalic acid can be safely substituted for metaphosphoric acid in determination of ascorbic acid, thus providing a more stable, more easily obtainable, and less expensive extractant.

\*Direct microscopic method to estimate sanitary history of frozen pack peas. E. R. Wolford. West. Canner and Packer 35(13):58. Dec., 1943. Enumerates advantages of direct test over plate-count or cultural method for frozen food products.

Determination of sulfur dioxide in dehydrated foods. A. N. Prater, C. M. Johnson, M. F. Pool, and G. Mackinney. (With Univ. Calif.) Indus. and Engin. Chem., Analyt. Ed. 16(3):153-157. March, 1944. The method proposed in this paper was designed as a control measure for inspection and field work with minimum equipment, but it is well adapted to laboratory research. It has been checked against other methods and also by the polarograph.

Rapid estimation of peroxidase in vegetable extracts--an index of blanching adequacy for frozen vegetables. M. P. Masure and H. Campbell. Fruit Prod. Jour. and Amer. Food Mfr. 23(12):369-374. Aug., 1944. A simple and rapid colorimetric method for the quantitative estimation of peroxidase activity in vegetable extracts. Values obtained show correlation with degree of blanch in stored vegetables preserved by freezing. The quantitative method has been modified to provide a simpler, semi-quantitative test for plant use.

Determination of crude lipid in vegetable matter. J. P. Nielsen and G. S. Bohart. Indus. and Engin. Chem. 16(11):701-703. Nov., 1944. Sweet corn and other vegetables increase in lipid content as they mature and a simple method for determination of crude lipid might serve as an index of maturity. Complete procedure including preparation of sample can be carried out in a short time. The method yields considerably larger quantities of crude lipid from certain types of vegetable material such as immature seeds than do the commonly accepted procedures for crude fat. It is equally well adapted to wet or dry, ground products.

Apparent increase in carotene of carrots during process of dehydration. G. F. Bailey and H. J. Dutton. Fruit Prod. Jour. and Amer. Food Mfr. 24(5):138-142. Jan., 1945. It was shown that observed apparent increase in carotene content is not a function of a systematic error in the carotene determination but is primarily a direct result of loss of soluble solids during processing.

\*Rapid determination of starch. Factors for starches and comparison with acid and enzymic hydrolysis methods. J. P. Nielsen and P. C. Gleason. Indus. and Engin. Chem., Analyt. Ed. 17(3):131-134. March, 1945. Presents factors for calculation of starch in various vegetables with potato starch as a standard, compares results by the proposed method with those by an enzyme procedure, describes minor revisions that increase accuracy of the original method (see above), and describes application to dehydrated foods. Data are presented to show that the enzyme procedure used for comparison is not applicable to all types of starches.

Specific heats of dehydrated vegetables and egg powder. F. Stitt and E. K. Kennedy. Food Res. 10(5):426-436. Sept., 1945. In the course of an investigation of heat transfer through dehydrated vegetables and egg powder, values of the mean specific heats of these materials between room and dehydrator temperatures were required. Mean specific heats of seven dehydrated vegetables and of egg powder were measured for the temperature interval 27 to 66°C. Measurements between 0 and 27°C. were also made on four of these materials. The heat capacity was found to exceed the sum of the heat capacities of the equivalent quantities of dry solid and water by an amount which varies with moisture content in different ways for dehydrated carrots, eggs, and potatoes. These positive deviations from additivity are discussed with regard to origin, thermodynamic significance, and interpretation in terms of "bound water" content.

Radio-frequency dielectric properties of dehydrated carrots. Application to moisture determination by electrical methods. W. C. Dunlap, and B. Makower. Jour. Phys. Chem. 49(6):601-622. Nov., 1945. Few comprehensive studies have been made of radio-frequency properties of vegetable materials containing absorbed water. This paper reports measurements made of the radio-frequency dielectric properties of dehydrated carrots. Dielectric constant and specific conductivity (a.c. and d.c.) as functions of moisture content (1.5-21.6 percent), temperature (1.2-39.8°C.), frequency (18 kc. to 5 mc.), density (0.760-1.38 gm per cubic centimeter), and particle size were measured. Use of variables, including direct-current conductivity, dielectric constant, impedance, dielectric loss, and power-factor measurements, for measurement of moisture content of organic materials containing adsorbed water is discussed. Although the d. c. conductivity is the most sensitive to moisture, its low average value and high sensitivity to temperature make it unsuitable for moisture determination. In general, the variables other than d. c. conductivity appeared to be nearly equivalent for moisture determination. In all cases except for dielectric constant at low moistures, a high radio-frequency is most suitable.

Seasonal variation in the enzyme content of eleven varieties of carrots. H. J. Morris, C. A. Weast, and H. Lineweaver. *The Bot. Gazette* 107(3): 362-372. March, 1946. Two harvests ( 2 months apart) were made of eleven varieties of carrots planted in August and March. Peeled portions were assayed for catalase, peroxidase, ascorbic-acid oxidase, and for those planted in March, also for phosphatase. Activities per gram of peeled tissue were about the same as for whole carrot root, except for peroxidase, which in samples tested was concentrated in the peel. Except for peroxidase, which varied little in average activity for all varieties, highest activities were found for carrots harvested in September, March, and January.

Structural composition of the sclereids in the integument of Pisum sativum L. R. M. Reeve. *Amer. Jour. Bot.* 33(3):191-204. March, 1946. Histochemical methods of detecting pentosans in the walls of "hemicellulosic" tissues in seed plants were developed by adaptation of reagents that yield characteristic color products of condensation with pentoses. Differentiation between lignin and pentosan reactions was accomplished. With these methods it was possible to detect pentosans in secondary wall thickenings of the macrosclereids and the osteosclereids in the integuments of peas and lima beans. Also, color differentiation between these components and the polyuronides of the middle lamellae was obtained.

The content and biological availability of carotene in dehydrated carrots stored at high temperatures. G. F. Bailey, H. J. Dutton, A. M. Ambrose, R. H. Wilson, and F. DeEds. *Arch. Biochem.* 10(1):125-130. May, 1946. Dehydrated carrots stored at 98°F. and 120°F. in an atmosphere of CO<sub>2</sub> were changed so as to be unacceptable from an aesthetic and organoleptic point of view, but carotene content was not lowered at 120°F. as much as during storage at 98°F. or lower. That partial destruction of carrots at elevated temperature created conditions unfavorable to the oxidation of carotene is suggested. Spectrophotometric analysis and bioassay of extracts agreed well. However, when solid material was used for bioassay, less than one-third of the carotene present was available to the rat.

The content and biological availability of carotene in raw and dehydrated carrots and other vegetables. R. H. Wilson, A. M. Ambrose, F. DeEds, H. J. Dutton, and G. F. Bailey. *Arch. Biochem.* 10(1):131-140. May, 1946. That chemical and bioassay methods for determining carotene do not agree, due to the inability of the test animal to adsorb all of the carotene in supplement, is the conclusion in this report. When carotene, which is poorly utilized in the dry product, is extracted and fed in cottonseed oil, the rat can use it. If knowledge of the carotene content of a sample is wanted, chemical assay would be the method of choice. If utilization of carotene is important question, chemical analysis may not give a true answer and the biological assay would be preferable. The carotene of dehydrated carrots is not always fully available to the albino rat. Of the other dehydrated vegetables studied, there was substantial agreement between the two methods of assay for carotene, indicating that the rat can utilize this carotene. The one exception was corn, where utilization was poor.

Presence of a growth-inhibiting substance in raw soybeans. A. A. Klose, B. Hill, and H. L. Fevold. Proc. Soc. Expt. Biol. and Med. 62(1):10-12. May, 1946. The low nutritional value of raw soybean proteins and marked improvement on heating have been recognized for some time; however, the fundamental mechanism by which this change takes place has not been satisfactorily explained. The experimentally demonstrated effect of heating is to make the methionine of protein nutritionally available. A substance which inhibits the growth-promoting properties of proteins in rats is present in raw soybeans. The substance is non-dialyzable, precipitated by salt, and inactivated by heat. It appears to be a protein, which has been shown to be concentrated in the acid-soluble (pH 4.2) fraction of soybean protein.

Moisture determination in dehydrated vegetables. Vacuum oven method. B. Makower, S. M. Chastain, and E. Nielsen. Indus. and Engin. Chem. 38(7): 725-731. July, 1946. The loss of weight when dehydrated potatoes, carrots, cabbage, and onions are heated in a vacuum oven has been studied as a function of time, temperature, fineness of grinding of sample, and degree of evacuation in the oven. The results were used for calibration of vacuum oven method against a primary reference method, which consisted of drying to constant weight in vacuo over magnesium perchlorate at room temperature. Since this method was inconveniently slow, a more rapid, but equivalent, secondary method was also established. The conditions for determination of water with an accuracy of  $\pm 0.1$  percent were as follows: pressure, 5 mm. of mercury or less; fineness of sample, ground to pass a 40-mesh sieve; time of heating, carrots at 70°, 29 to 35 hours, potatoes at 70°, 43 to 67 hours, cabbage at 60°, 22 to 40 hours, and onions at 60°C., 15 to 45 hours.

#### Dehydration

##### Mimeographed circulars:

- AIC-15 Bin-type finishing driers in vegetable dehydration. 1943. Revised July, 1944.
- AIC-31 Application of drying rate nomographs to the estimation of tunnel-dehydrator drying capacity.
- I. Riced white potatoes. Nov., 1943. Revised June, 1947.
  - II. Blanched sweet corn. Nov., 1943
  - III. White potato strips--vertical air flow. Jan., 1944.
  - IV. Shredded cabbage. Feb., 1944.
  - V. Onion slices. April, 1944.
  - VI. Sweet potato strips. Sept., 1944.
  - VII. White potato half cubes. March, 1945.
  - VIII. Carrot pieces. May, 1947.
- AIC-35 Determination of ascorbic acid in fresh, frozen, and dehydrated foods. Dec., 1943.
- AIC-39 Cost accounting for vegetable dehydration plants. Jan., 1944.
- AIC-47 The sampling and analysis of gases in cans of dehydrated vegetables. June, 1944.
- AIC-58 Peroxidase test procedure for dehydrated potatoes to indicate adequacy of blanching. Aug., 1944.

- AIC-75 Tray materials in relation to sulfited, dehydrated vegetables. March, 1945.
- \*AIC-152 Approximate diffusion calculations for the falling-rate phase of drying. April, 1947.

Dehydrator designs:

- Type A - Transverse - flow cabinet dehydrator.
- Type I - Steam-heated cabinet dehydrator (single-truck unit).
- Type J - Steam-heated cabinet dehydrator (double-truck unit).
- Type N - Vegetable dehydrator, tunnel type, two-stage.
- Type O - Cabinet dehydrator with cabinet blancher and bin finisher.
- Types P, Q - Counter flow tunnel dehydrators.
- Dwg. D-96 - Multibin finisher.
- Dwgs. C-112, 113 - Laboratory experimental cabinet drier.
- Dwg. C-115 - Steam heating arrangements for tunnel dehydrators.
- Dwg. A-118 - Friction stop for trucks.

Preparation-equipment designs:

- Dwg. D-108 - Tray-loading and de-traying table.
- Dwg. D-116 - Picking and trimming table.
- Dwg. D-214 - Draper-type-atmospheric steam blancher.

Bulletins:

Preservation of fruits and vegetables by commercial dehydration. U. S. Dept. Agr. Circ. 619. 46 pages. Feb., 1942.

Commercial dehydration of vegetables and fruits in wartime. U. S. Dept. Agr. Misc. Pub. 524. 29 pages. Sept., 1943.

\*Vegetable and fruit dehydration. A manual for plant operators. U. S. Dept. Agr. Misc. Pub. 540. 218 pages. June, 1944.

Journal articles

Present status of dehydration in the United States. E. H. Chace. Proc. Inst. Food Technol. pp. 70-89. 1942. Brief account of dehydration investigations initiated by Chief of Bureau of Agricultural Chemistry and Engineering at beginning of World War II. Conditions and practices required for operating a drying plant are reviewed.

\*Tunnel dehydrators and their use in vegetable dehydration. W.B. Van Arsdel. Food Indus. I. 14(19):43-46, 106. 1942; II. 14(11):47-50. 1942; III. 14(12):47-50. 1942. Factors governing choice of dehydrator for vegetables, various available arrangements of tunnel dehydrators, physical laws and engineering fundamentals of dehydrator operation, and operating characteristics of tunnel dehydrators are discussed.

\*Vitamin A value of fresh and dehydrated carrots. R. H. Wilson, J. O. Thomas, and F. DeEds. Fruit Prod. Jour. 22(1):15-17. Sept., 1942. Concerns vitamin A value of fresh and dehydrated carrots of the Chantancy variety. The parallelism between chemical and biological methods for evaluating vitamin A both before and after a processing technique is studied.

Facts of vegetable dehydration revealed by microscope. R. M. Reeve. Food Indus. 14(12):51-54. Dec., 1942. Physical and some chemical changes were

studied microscopically. The observations show correlations with some of the analytical data on vitamin retention, and help to explain some of the reasons for vitamin loss.

\*Packaging requirements for dehydrated vegetables. A. L. Pitman, W. Rabak, and H. Yee. Food Indus. 15(1):49-52. Jan., 1943. What a package must do to protect quality of dehydrated vegetables is explained. Authors give relative performance of various materials as determined by tests.

\*A microscopic study of physical changes in carrots and potatoes during dehydration. R. M. Reeve. Food Res. 8(2):128-136. Mar.-April, 1943. Preparation of vegetable tissues for microscopic examination after different periods and techniques of drying was accomplished by a modified celloidin technique, by which dehydrated condition was preserved. The extent of shrinkage during dehydration, and differences between blanched and unblanched dehydrated carrots and potatoes are described.

Effects of varying conditions on the reconstitution of dehydrated vegetables. M. E. Davis and L. B. Howard. Proc. Inst. Food Technol., pp. 143-155. 1943. The implications of various terms applied to the process of replacing water in dehydrated vegetables are discussed. Some of the changes during this process can be measured quantitatively and expressed mathematically. Acceptable techniques in rehydration tests must provide for: (1) time and temperature sequences suitable to material and compatible with optimum quality; (2) enough water to submerge pieces without excessive amounts at end of test; (3) shaking or stirring when necessary to insure wetting of all pieces; (4) control of rate of heating so as to prevent rapid and variable losses of water during boiling; and (5) relatively pure water of constant composition. The authors discuss relationship between blanching and rates of rehydration and cooking, and effects of varying the temperature, time of drying, and lowering or raising moisture content in finished dried carrots.

This is what it costs to dehydrate vegetables. W. D. Ramage and C. L. Rasmussen. Food Indus. I. Buildings, plant layout, capital investment. 15(7):64-71. July, 1943; IIA. Processing costs--labor, raw material. 15(8):66-67. Aug., 1943; IIB. Processing costs--summarized. 15(9):75-77. Sept., 1943. Plant layout, capital investment, and operating costs are given for plants of 25 to 100 tons daily capacity. Layouts are illustrated and operating practices discussed. Processing costs are discussed in detail in the second and third parts of the article.

\*Some engineering problems of the new vegetable dehydration industry. W. B. Van Arsdel. Heating, Piping and Air Conditioning 15(3):157-160. 1943.: Wartime conditions introduced a number of new dehydration systems for drying of foods. Six essential specifications to be considered in design of a dehydration system and complete new data on vegetable dehydration with charts showing drying rates are provided for use of plant operators. Emphasis is placed upon design of equipment that requires fewer man hours of skilled labor in fabricating equipment.

\*Tray and tunnel drying methods and equipment. W. B. Van Arsdel. Proc. Inst. Food Technol., pp. 45-51. 1943. Certain aspects of driers and drying methods with special reference to tray and tunnel drying are discussed. Kinds of jobs vegetable driers perform are classified. A definition of a good tray drier is provided; counterflow tunnel driers are

discussed; and brief comment on probable evolution of design as applied to tray and tunnel driers is given.

Drying characteristics of vegetables-riced potatoes. A. H. Brown and P. W. Kilpatrick. Trans. Amer. Soc. Mech. Engin. 65(11):837-842. Nov., 1943. Experimental data upon which basis dehydrators could be designed and built. Equipment involved, conditions of the investigation as applied to riced potatoes, and analysis of experimental results led to presentation of a rational theory of dehydrator design and operation.

\*Dehydrated spinach. Changes in color and pigments during processing and storage. H. J. Dutton, G. F. Bailey, and E. Kohake. Indus. and Engin. Chem. 35(11):1173-1177. Nov., 1943. Experiments have shown that, in unblanched dehydrated spinach, destruction of chlorophyll is positively correlated with moisture content and is affected very little by oxygen content of the storage atmosphere. Destruction of carotene is greatly dependent upon oxygen in the storage atmosphere and independent of moisture content of product. Destruction of chlorophyll may be regarded as indication of loss of palatability and ascorbic acid, but does not indicate loss of carotene. Because changes in color due to changes in chlorophyll obscure changes in color due to changes in carotene content, there is little possibility of correlating carotene content with spectral reflectance.

\*Cabinet dehydrators suited to small-scale operations. E. A. Beavens. Food Indus.: I. 16(11):70-72. Jan., 1944; II. 16(2):90-92. Feb., 1944; III. 16(3):75. Mar., 1944. These units are practicable for experimental work and for dehydrating from 1 to 20 tons of fresh vegetables a day. They also permit handling of different products at the same time. The author discusses types of cabinets and their operation and uses. Cabinet dehydrators have definite advantages for certain operations. Single cabinets are practicable for experimental work. Multistage cabinet units, combination compartment-and-tunnel systems and duplex cabinet arrangements are used for commercial operation.

Sulphite retards deterioration of dehydrated cabbage shreds. G. Mackinney and L. B. Howard. Food Indus. 16(5):355-356. May, 1944. (With Univ. Calif.). An economically feasible and practical method of sulphiting was sought which could be readily applied by plants working under severe wartime handicaps with respect to equipment and personnel. Presents specific example of such a method and value of process with respect to retarding deterioration of a dehydrated product.

\*Significance of moisture content of dehydrated vegetables. L. B. Howard. Canner 100(3):46. Feb., 1945. Reasons for dehydrating to low moisture levels, methods for determining moisture content and keeping quality of dehydrated products, tests to determine deterioration, and a description of "in-package" desiccation are presented.

Severe blanch doesn't improve dehydrated potato quality. H. Campbell, H. Lineweaver, and H. J. Morris. Food Indus. 17(4):384-386. April, 1945. Blanching white potatoes prior to dehydration improves storage and rehydration characteristics. However, storage quality is not improved by severe blanching or by completely inactivating peroxidase enzyme system.



Residual peroxidase is of little moment. Potatoes blanched 10 minutes had no better keeping quality than potatoes blanched 1 or 2 minutes. Below 70°F., temperature does not markedly affect rate of deterioration. A storage temperature as high as 90°F. for long periods is undesirable, especially from standpoint of color change. Some lots are more subject to overblanching or so-called "popcorn" effect than others. Popcorn effect appears to be related to degree of mealiness in stock.

\*Some fundamentals of vegetable preservation by freezing. H. Cambell. West. Frozen Foods 6(8):3-5. June, 1945. Emphasizes necessity for high and uniform levels of quality if progress made in vegetable preservation by freezing is to be maintained. The author discusses raw materials from standpoint of variety, source, maturity, and handling. Under processing methods he treats blanching, cooling, and the freezing procedure. Storage practices are discussed with reference to temperature and stacking. The hazards are pointed out and means of avoidance are provided.

\*Commercial sulfiting practices. E. A. Beavens and J. A. Bourne. Food Indus. 17(9):100-101. Sept., 1945. Dehydration plants in Southern California treat carrots and cabbage with sulphite solutions to retain flavor, color, and vitamin content. A continuous sulphiting line for treating vegetables before blanching is used in these plants. The solution is fed to the spray from an overhead steam-jacketed kettle equipped with an electronic level controller. It is pumped into this kettle from two storage tanks equipped with agitators, passing through a filter after it leaves the stainless-steel pump.

Specific heats of dehydrated vegetables and egg powder. F. Stitt and E. K. Kennedy. Food Res. 10(5):426-436, Sept.-Oct., 1945. Mean specific heats of seven dehydrated vegetables and of egg powder were measured for temperature interval of 27° to 66°C. Measurements between 0° and 27°C. were also made on four of these materials. Heat capacity was found to exceed the sum of the heat capacities of the equivalent quantities of dry solid and water by an amount which varies with moisture content in different ways for dehydrated carrots, eggs, and potatoes. These positive deviations from additivity are discussed with regard to origin, thermodynamic significance, and interpretation in terms of "bound water" content.

\*Recent engineering investigations in food dehydration at the Western Regional Research Laboratory. W. B. Van Arsdell. Proc. 4th Ann. Meet. National Dehydrators Assoc. pp. 33-35. 1945. Brief report on certain phases of engineering work on dehydration. Removal of moisture to low ranges by tunnel dehydrators having proved rather ineffective, use of finishing bins as a means of reducing moisture content is discussed.

Effect of storage temperatures on sensitivity of White Rose potatoes to processing heat. H. Campbell and P. W. Kilpatrick. Fruit Prod. Jour. and Amer. Food Mfr. 24(4):106-108. Dec., 1945. In commercial and experimental dehydration of white potatoes, considerable variation has been observed between lots with respect to sensitivity to heat damage during dehydration under similar conditions. Heat damage is characterized by

reddening or browning of dried tissue, especially in centers of strips and cubes. Variability between lots of commercially and experimentally dehydrated potatoes with respect to sensitivity to heat when dehydrated can be attributed in part to storage of raw stock at temperatures that permit accumulation of sugars. The sensitivity of potatoes to heat damage resulting from low-temperature storage can be reduced by subsequent storage at elevated temperature prior to dehydration.

\*Progress in spray drying of fruits and vegetables. J. R. Matchett. West Canner and Packer 38(1):71-72. Jan., 1946. Spray drying is a process designed to desiccate almost instantly products which would be harmed by prolonged exposure to high temperatures or those whose characteristics do not lend themselves well to other means of dehydration. The process of spray drying, which is used extensively for dehydration of milk and eggs, may prove to be well adapted to removal of moisture from other fluid food products that are easily injured by heat. In this process dehydration is accomplished within a few seconds by suspending finely atomized fluid in a stream of hot air and collecting the resulting dry product as a powder.

Dehydrofreezing -- new way of preserving food. L. B. Howard, and H. Campbell. Food Indus. 18(5):674-676. May, 1946. Experiments with several fruits and vegetables have shown that moderate dehydration (removal of about two-thirds of water content) followed by freezing storage resulted in excellent products in most cases. Advantages of this new method are savings in weight, space, packaging materials, and refrigeration load. The preliminary experiments have dealt with potatoes, peas, carrots, asparagus, cherries, boysenberries, and apricots.

+Vacuum drying of compressed vegetable blocks. W. C. Dunlap. Indus. and Eng. Chem. 38(12):1251-1253. Dec., 1946. Drying compressed blocks of partially dried potato and carrot by use of vacuum and radio frequency heat at 60°C. was compared with drying vacuum oven at 60°C. Although radio-frequency method had definite advantages with respect to speed of drying, vacuum-oven method produced higher quality. Vacuum oven set at 70°C. appeared to be roughly equivalent, with respect to speed of drying and quality, to radio-frequency method at 60°C.

+Approximate diffusion calculations for the falling-rate phase of drying. W. B. Van Arsdel. Chem. Engin. Progress 43(1):13-24. Jan., 1947. Approximate numerical procedures are applied in the present paper to explore the effect of a variable diffusivity in the low-moisture range upon the shape of rate curve for the infinite slab, the long cylinder, and the sphere. Either vapor pressure or concentration may be used as the driving potential. The effects of changing thickness of body and changing relative importance of surface mass transfer coefficient are investigated. Typical experimental rate curves, determined during dehydration of cut vegetables, exhibit curvature of type predicted by such calculations, and to a degree that might well be expected on the basis of independent measurements of diffusivity in hydrophilic solids.

\*Browning of dehydrated vegetables during storage. R. R. Legault, W. F. Talburt, A. M. Mylne, and L. A. Bryan. Indus. and Engin. Chem. 39(10): 1294-1299. Oct., 1947. The rate of nonenzymatic browning of dehydrated vegetables has been studied as a function of temperature, moisture content, and confining atmosphere. Browning proceeds in linear fashion up to, and for a reasonable distance beyond, the limit of palatability for dehydrated nonsulfited carrot, white potato, onion, and sweet potato. The effect of oxygen on browning rates of these four dehydrated vegetables is small. Browning rates vary exponentially with reciprocal of absolute temperature. Temperature coefficients are high, the  $Q_{10}$  values varying from 5.0 to 8.4 among these vegetables. Browning rates vary exponentially with moisture content over the range studied.



## WHEAT GLUTEN

### Industrial Utilization

AIC - 119 Glutamic acid review and bibliography, H. S. Olcott and G. H. Brother, Aug., 1946. Occurrence, properties, methods of preparation, and uses of glutamic acid and monosodium glutamate are briefly reviewed. The bibliography is divided with regard to subject matter and contains about 200 references.

Wheat gluten as an industrial protein. H. S. Olcott and M. J. Blish, Amer. Assoc. Cereal Chem. Trans. 2(2):20-25. Jan., 1944. A review of information regarding properties and possible uses of gluten.

A method for the determination of glutamic acid in proteins. H. S. Olcott Jour. Biol. Chem. 153(1):71-82. Apr., 1944. The method reported is based on the fact that glutamic acid can be converted to pyrrolidonecarboxylic acid under conditions that do not affect other amino acids, except cystine and cysteine. The corresponding decrease in amino nitrogen, corrected for cystine, can be used to estimate the amount of glutamic acid in protein hydrolysates. By the use of the Van Slyke manometric apparatus, the method is applicable to the determination of glutamic acid in 25 to 100 mg. samples of protein with an estimated accuracy of 5 percent. The glutamic acid is approximately 92 percent converted in 4 hours. Of a number of amino acids investigated only cystine interferes. The glutamic acid content of a number of proteins was determined; most results agreed with previous reports. Insulin contained approximately 20 percent of glutamic acid; gelatin, 12 percent; glutenin, 36 percent; chicken feathers, 12 percent; cottonseed globulin, 20 percent; and tobacco mosaic virus, 17 percent.

Outlook for wheat gluten as an industrial material. H. S. Olcott, Food Indus. 16(7):541, 576-578. July, 1944. Overall picture is given--source of supply, methods of manufacture, properties and potential industrial uses. Possibilities as a food material are shown to be dependent upon economic supply. Wheat gluten, a standard but limited article of commerce for several decades, has long been used in special diabetic foods. Its chief use in recent years has been as a source of monosodium glutamate, a flavoring or condiment.

\*Gel-forming derivatives of wheat gluten. H. C. Reitz, R. E. Ferrel, and H. S. Olcott, Indus. and Eng. Chem. 36(12):1149-1151. Dec., 1944. A product obtained from wheat gluten by the action of chlorosulfonic acid and pyridine or cold concentrated sulfuric acid possesses, after neutralization, the property of absorbing rapidly one hundred to three hundred times its weight of cold water to form a firm, odorless, tasteless, and nontoxic gel. The use of gluten sulfate as a substitute for scarce natural gums in therapeutic jellies, ointments, and other pharmaceutical preparations, and as a thickening agent or emulsifying agent in ice cream or other foods, is suggested.

\*A lactobacillus assay method for p-aminobenzoic acid. J. C. Lewis, Jour. Biol. Chem. 146(2):441-450. Dec., 1942. A microbiological assay method for p-aminobenzoic acid is described. The method is based on the growth

factor activity of p-aminobenzoic acid for Lactobacillus arabinosus. Its reliability is indicated by reproducibility of assays, by agreement of assay values obtained with varying levels of added sample, and by recoveries of p-aminobenzoic acid added to various samples. The method is very sensitive, the assay range being 0.15 to 0.5 millimicrogram of p-aminobenzoic acid. Assay values for a number of biological materials are included. Occurrence of biologically inactive forms of p-aminobenzoic acid is indicated. These are activated by alkaline hydrolysis.

\*A Lactobacillus assay method for l(+) - glutamic acid. J. C. Lewis and H. S. Olcott. Jour. Biol. Chem. 157(1):265-285. Jan., 1945. This paper presents a quantitative method for the determination of l(+)-glutamic acid based upon its ability to stimulate growth of this organism, as measured by production of lactic acid, in a medium containing known requirements with the exception of l(+) -glutamic acid. The techniques are based for the most part on those previously described for the microbiological determination of vitamins and amino acids by the use of Lactobacillus arabinosus.

### Properties

Stability of wheat gluten dispersions toward reducing agents in the presence and absence of a gluten proteinase. H. S. Olcott, L. A. Sapirstein, and M. J. Blish. Cereal Chem. 20(1):87-97. Jan., 1943. Gluten dispersions in dilute acetic acid can be stabilized by heating for 5 to 10 minutes at 100°C. The solubility characteristics of gluten proteins are not appreciably altered, but an enzyme (proteinase) responsible for instability is destroyed. Proteinase is inactivated or inhibited in 10 percent sodium salicylate but activity is restored and proteinase-induced hydrolysis continues if salicylate is removed. Reducing agents increase enzymatic hydrolysis slightly but drastically reduce the viscosity of gluten freed from enzyme activity by heat or by the presence of 10 percent sodium salicylate. That the primary effect of reducing agents on gluteins is a chemical one upon proteins and only secondarily that of enzyme activation, is the conclusion reached, and it is suggested that a similar explanation can be applied to effect of reducing agents, but not necessarily oxidizing agents, on doughs.

+Research on wheat gluten. G. H. Brother and H. S. Olcott. Bakers Digest 21, 97. Oct., 1947. A brief review.

+Characterization of wheat gluten. I. Protein-lipid complex formation during doughing of flours. Lipoprotein nature of the glutenin fraction. H. S. Olcott and D. K. Mecham. Cereal Chem. 24(6):407-414. Nov., 1947. A high-protein patent flour was found to contain 1.5 percent total lipids, 70 percent of which were extractable with ether. The flour was mixed with water with minimum doughing, then dried by lyophilization. Only 40 percent of the lipids were now extractable. After the flour was kneaded into dough and dried, less than 10 percent of the lipids could be extracted. The capacity of the flour to "bind" lipids during wetting and doughing was ascertained by determining the extractability of added flour lipids. At least three times the amount of lipid normally present could be bound by the doughing procedure. Phospholipids were bound preferentially. Most of the lipid bound was associated with the gluten

rather than nonprotein constituents; and, when gluten was fractionated the lipid was found to be bound to the "glutenin" rather than the gliadin fraction. Glutenin fractions containing up to 20 percent lipids have been obtained. It is proposed to call the nongliadin fraction of gluten "lipoglutenin". The term "glutenin" should be reserved for fat-free preparations.

### Chemical Modification

Monothioglycol. H. S. Olcott. Science 96(2498):454. Nov., 1942. This paper presents useful data on monothioglycol,  $\text{CH}_2\text{OHCH}_2\text{SH}$  (also designated thioglycol, monothioethylene glycol, and beta - or 2-mercaptoethanol); the compound is a useful non-nitrogenous sulfhydryl reagent for protein investigations.

The action of 1,2-epoxides on proteins. H. Fraenkel-Conrat. Jour. Biol. Chem. 154(1):227-238. June, 1944. Epoxides, such as ethylene oxide, propylene oxide, and epichlorohydrin, were found to be suitable reagents for the esterification of protein carboxyl groups in aqueous solution at room temperature. Sulfhydryl, amino, and phenolic groups also reacted. Through treatment of crystalline egg albumin and beta-lactoglobulin with these compounds, preparations of modified protein were obtained that differed from the original material in several particulars. The isoelectric point was increased from 2 to 3 pH units. In general, the newly introduced bonds were surprisingly stable in acid and alkaline solution with the exception of small proportions of the substituted carboxyl and amino groups, which were hydrolyzed readily in acid and alkali, respectively.

Action of aromatic isocyanates on proteins. H. Fraenkel-Conrat, M. Cooper, and H. S. Olcott. Jour. Amer. Chem. Soc. 67(21):314-319. Feb., 1945. Aromatic isocyanates in anhydrous media are widely used for the characterization of alcohols and amines. This paper reports reaction of proteins when treated with aromatic isocyanates under anhydrous conditions and in presence of pyridine. Reaction occurred with: (1) basic groups, including amino, guanidyl, and imidazole; (2) acid groups, including carboxyl, thiol, and phenolic; and (3) primary amide and probably part of the aliphatic hydroxyl groups. Under the same conditions the peptide groups of chain molecules did not react appreciably, in contrast to the simple diketopiperazines which bind two molecules of phenyl isocyanate. The method described by Plimmer for quantitative liberation of the primary amide, amino, and the guanidyl nitrogen by the action of nitrous acid in mineral acid was applied successfully to proteins. This reaction made possible estimation of the extent of interaction of amide groups with phenyl isocyanate. The products of the anhydrous reaction of phenyl isocyanate with alanine and with glycine and alanine anhydrides are described.

The reaction of formaldehyde with proteins. H. Fraenkel-Conrat, M. Cooper, and H. S. Olcott. Jour. Amer. Chem. Soc. 67(6):950-954. June, 1945. The reaction of formaldehyde with proteins is of importance in a number of fields, some of which are tanning of leather, hardening of tissues, fibers, and plastics, and preparation of toxoids. This paper reports that in 4 percent solution, at pH 3 to 7 and  $70^\circ$ , formaldehyde combined with both the primary amino and the primary amide groups of proteins. In

contrast to interpretation of other investigators, the secondary amide linkages of the peptide chain, and the phenolic groups, were found not to bind appreciable amounts of formaldehyde. These findings were confirmed with protein derivatives and synthetic polypeptides containing a maximal or minimal number of reactive groups. The preparation of a polypeptide rich in primary amide groups from polyglutamic acid is described. This material bound more formaldehyde than did any of the proteins investigated.

Esterification of proteins with alcohols of low molecular weight. H. Fraenkel-Conrat and H. S. Olcott. Jour. Biol. Chem. 161(1):259-268, Nov. 1945. Carboxyl groups of proteins and model substances are readily methylated at room temperature in methyl alcohol containing small amounts of mineral acids (0.02 to 0.1N). The catalytic activity of acid chlorides in esterifications is due to the liberation of hydrochloric acid. The acid-catalyzed reaction of proteins with methanol appears to be a specific one involving only the carboxyl groups; amino, phenolic, thiol, and indole groups and peptide and amide bonds are unaffected. Esterification occurs also with higher primary alcohols, but is slower and progressively less complete than with methanol. Several proteins dissolve in the alcohols during the reaction.

Reaction of formaldehyde with proteins. II. Participation of the guanidyl groups and evidence of crosslinking. H. Fraenkel-Conrat and H. S. Olcott. Jour. Amer. Chem. Soc. 68(1):34-37. Jan., 1946. The reaction of formaldehyde with amino acids and proteins has received renewed attention and application in recent years. This paper reports that guanidyl groups bind formaldehyde at 70° in neutral or acid solution. Protamine sulfate and simple substituted guanidine salts bind up to 2 moles for each guanidine group, free guanidine salts up to 3 moles of formaldehyde. At room temperature the reaction proceeds slowly and to a lesser extent. The reactivity of the guanidyl nitrogen toward nitrous acid in mineral acid is decreased after combination with formaldehyde. A decrease in the basicity of the guanidyl groups is also evident. If the reaction is performed at protamine concentrations exceeding 3 percent, the product is largely water insoluble. In 1-3 percent solution and at pH 6-7, the reaction product remains soluble, but differs from untreated protamine in being in part nondialyzable. Osmotic pressure measurements indicate considerable increase in the average molecular weight of these treated preparations. This is regarded as evidence for intermolecular condensation through crosslinking methylene groups.

Water resistance of proteins. Improvement through chemical modification. H. S. Olcott and H. Fraenkel-Conrat, Indus. and Engin. Chem. 38(11):104, 106. Jan., 1946. Use of proteins in plastics is seriously restricted by poor water resistance. Proteins were treated with a number of organic reagents and the products were examined for water resistance by measurement of the uptake of water by pressed disks. Aryl and long chain alkyl isocyanates and also aromatic acid anhydrides and chlorides proved most effective. A number of proteins yielded phenyl isocyanate derivatives showing 24-hour water absorption of 1 to 2 percent. Phthalic anhydride gave products of low water absorption with egg white and cattle hoof. Protein derivatives of low water absorption showed a tendency to plastic flow without the addition of water as plasticizer.



Action of sulfating agents on proteins and model substances. I. Concentrated sulfuric acid. H. C. Reitz, R. E. Ferrel, H. Fraenkel-Conrat, and H. S. Olcott, Jour. Amer. Chem. Soc. 68(6):1024-1031, June, 1946. The reaction of cold, concentrated sulfuric acid on proteins resulted in the approximately quantitative formation of acid sulfates on the hydroxyl groups of serine, threonine, and hydroxyproline residues. The sulfhydryl group of cysteine reacted to form the thiosulfate. Under the specific conditions used, hydroxyl groups of tyrosine were not appreciably sulfated; some ring sulfonation occurred. The O-sulfuric acid esters of serine, threonine, and hydroxyproline, and S-cysteine sulfonate were prepared by the action of concentrated sulfuric acid on the amino acids. The stabilities of the protein and amino acid sulfate bonds in sulfuric acid and in dilute acid and alkaline solutions were determined.

Action of sulfating agents on proteins and model substances. II. Pyridine-chlorosulfonic acid. H. C. Reitz, R. E. Ferrel, H. S. Olcott, and H. Fraenkel-Conrat, Jour. Amer. Chem. Soc. 68(6):1031-1035. June, 1946. Various proteins were treated with the reaction product of chlorosulfonic acid and pyridine under anhydrous conditions at 70-80° for two and one-half hours. This led to the covalent binding of considerable amounts of sulfate sulfur (up to 10 percent), corresponding in several proteins to the sulfation of one out of every three amino acid residues. The mode of linkage was ascertained through the use of model substances and proteins or polypeptides rich in certain groups. It was thus shown that, under the condition used, part or all of the aliphatic and phenolic hydroxyl, thiol, primary amide, amino, guanidyl and indole groups were transformed to sulfates or sulfamates. For most proteins, there was definite correlation between the sum of these groups and the sulfate introduced. The imidazole and carboxyl groups and the peptide linkage did not participate in the reaction. The stability of the various sulfate linkages in acid and alkali was investigated. Sulfamates of indoles or guanidines appear not to have been described previously.

\*Specific group reagents for proteins. H. S. Olcott and H. Fraenkel-Conrat. Chem. Rev. 41(1):151-197. Aug., 1947. A detailed review of protein groups and the reagents available to substitute each of the different kinds specifically. Tables summarize the information available up to the time of publication.

\*Effect of dry heat on proteins. D. K. Mecham and H. S. Olcott. Indus. and Engin. Chem. 39(8):1023-1027. Aug., 1947. Wheat gluten, casein, zein, egg white, cattle hoof, and soybean protein were heated in boiling inert hydrocarbons at temperatures from 110° to 203°C. for 18 hours. Solubility decreased markedly with increase of heating temperature up to 153°, but there was no change in total nitrogen and little change in the amide nitrogen contents. Above 153° extensive degradation occurred, with continuing loss of water and the formation of more soluble products. With wheat gluten and zein, total and amide nitrogen contents were decreased. The amino and total basic groups of all proteins were found to be decreased by the heat treatment. Wheat gluten and cattle hoof, after being heated above 153°, were not digestible by pancreatin. The cystine content of cattle hoof was markedly decreased by heat treatment above 153°. Equilibrium moisture contents at 70 percent relative humidity

were decreased by heat treatment at all temperatures. The equilibrium moisture contents continued to decrease at temperatures above 153° despite the increasing solubility of the heated products.

### Methods

Effect of light on the Van Slyke method for the determination of amino groups. H. Fraenkel-Conrat. Jour. Biol. Chem. 148(2):453-454. May, 1943. During a study of protein-aldehyde reactions, unexpected variations were obtained in amino nitrogen determinations of proteins and their derivatives by the manometric method of Van Slyke. A search for the cause indicated a correlation between these high values and the intensity of light falling upon the reaction chamber. Further experiments indicated that tyrosine and its derivatives give off extra nitrogen under the conditions of the Van Slyke analysis when the reaction chamber is illuminated.

The determination of cysteine and cystine by Vassel's method. D. K. Mecham. Jour. Biol. Chem. 151(2):643-645. Dec., 1943. Increased precision in the determinations of cysteine and cysteine plus cystine, particularly in the presence of detergents, was obtained by modification of some details of Vassel's procedure.

\*The use of dyes for the determination of acid and basic groups in proteins. H. Fraenkel-Conrat and M. Cooper. Jour. Biol. Chem, 154(1): 239-246. June, 1944. Microanalytical methods developed for the estimation of the number of acid and basic groups of proteins are reported in this paper. These were based on the tendency of the polar groups to bind dyes of the opposite charge, resulting in a precipitation of the protein-dye complex. The acid dye, orange G, combined stoichiometrically with basic protein groups at pH 11.5, but the extent of combination was in this case slightly affected by other factors, such as protein concentration. The number of protein groups binding these dyes corresponded well to the total number of basic (guanidyl, imidazole, amino) and acid (carboxyl, phenol, thiol) groups of crystalline egg albumin and beta-lacto - globulin and, approximately, to those of several crude proteins studied. The proposed micro methods were applicable to both soluble and insoluble proteins. They have proved useful tools in the interpretation of the action of various chemical agents on proteins.

\*Reaction of formaldehyde with proteins. IV. Participation of indole groups. Gramicidin. H. Fraenkel-Conrat and H. S. Olcott. Jour. Biol. Chem. 168(1):99-118. April, 1947. Gramicidin binds rapidly in alkaline solution an amount of formaldehyde equivalent to its tryptophan content. In acid solution the reaction proceeds only at high temperature and formaldehyde concentrations. The formaldehyde adds as methylol groups probably to the nitrogen of the indole rings. Formaldehyde is released only partially during acid hydrolysis, but completely and without damage to the gramicidin molecule in strong alkali. The chromogenic activity of the tryptophan residues is largely abolished through combination with formaldehyde and regenerated by strong alkali. The tryptophan residues of proteins appear to react with formaldehyde

under the same conditions and in a manner similar to those of gramicidin. At room temperature there is little reaction at pH 7 to 8, even at high formaldehyde concentration, but at pH 11 the reaction is completed rapidly, even at low formaldehyde concentration. All 3-substituted indole derivatives, including proteins rich in tryptophan, bind formaldehyde in boiling N to 20N sulfuric acid. Simple 3-substituted indoles bind up to 1 mole of formaldehyde at room temperature, both in alkaline and in acid solution. 2,3-Dimethylindole reacts incompletely, even at elevated temperature. 1,3-Dimethylindole reacts appreciably only in acid yielding methylene bis (1,3-dimethylindole). Pure methylol derivatives were obtained from skatole and 2,3-dimethylindole. 1,2-Dimethylindole-3-acetic acid does not react with formaldehyde.

•Formation and loss of cysteine during acid hydrolysis of proteins. Role of tryptophan. H. S. Olcott and H. Fraenkel-Conrat. Jour. Biol. Chem. 171(2):583-594. Dec., 1947. Under the conditions usually used for acid hydrolysis of proteins (heating to 100° or 125° for 6 to 24 hours), cystine reacts with tryptophan to give cysteine. The reaction occurs much more readily in 6 to 7N hydrochloric acid than in 6N sulfuric acid. Some cysteine results from the acid hydrolysis of cystine in the absence of tryptophan. The reaction, however, is only appreciable at 125° in 6N sulfuric acid. The cysteine contents of acid hydrolysates of proteins containing no sulfhydryl groups reflect both of these reactions. Cysteine reacts with pyruvic acid in acid solution. This reaction may explain the loss of cysteine from protein acid hydrolysates noted by Halwer and Nutting. The oxidation of cysteine in hydrochloric acid solution is catalyzed by copper salts. Tryptophan is fairly stable in hot acid solutions in the absence of air. Both pyruvic acid and cystine cause destruction; the latter reaction occurs without formation of humin. The bearing of these observations on interpretation of amino acid composition of proteins is noted.

#### Patents

Proteinous compositions and their process of preparation. H. C. Reitz. No. 2,344,267. Patented March 14, 1944. Relates to preparation of sulphated proteins, particularly sulphated gluten. Process involves reacting protein with chlorosulphonic acid. Products have gelling properties and are valuable as substitutes for natural gums.



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General

Supplies of general information on the program of the Western Regional Research Laboratory and the Bureau of Agricultural and Industrial Chemistry are available.

Research on agricultural products. T. L. Swenson. Scientific Monthly 62(6): 525-537, June, 1946. Two examples of research in the Western Regional Laboratory are used to illustrate use of basic knowledge in the scientist's attempt to make something new and useful: Theoretical aspects of the work and fundamental facts and theories are described. One example (fibers from keratin) is essentially biochemical; the other (dielectric applications) is essentially biophysical.

Apparatus

Modification of Cenco spectrophotometer, permitting measurements of reflection and fluorescence spectra. H. J. Dutton and G. F. Bailey. Indus. and Engin. Chem., Analyt. Ed. 15(4):275-277, April, 1943. A simple modification of the Cenco spectrophotometer permits measurement of reflection and fluorescence spectra with moderate resolution. The arrangement whereby fluorescence is observed from the same direction as the exciting light is superior to other possible arrangements, in that shape of curve representing spectral distribution of energy and position of maximum are independent of concentration of fluorescing substance.

Recovery of agar from used media. A. A. Anderson. Jour. Bact. 46(4):396-397, Oct., 1943. Reports a simple and satisfactory method for recovery of agar from used culture media. Steps involved are: (1) drying, (2) grinding, (3) washing, and (4) drying.

A useful tool for shaping spectrographic graphite electrodes. E. J. Eastmond. Jour. Optical Soc. Amer. 34(10):621-622, Oct., 1944. Graphite electrodes with a central post and annular ring have been used in direct current arc methods of spectrographic analysis. Size of this cavity is dependent on form of sample under investigation and amount found necessary to give spectra suitable for the analysis required. A tool for shaping these graphite electrodes is described.

A simple automatic media dispenser. A. A. Andersen. Indus. and Engin. Chem., Analyt. Ed. 17(7):463-464, July, 1945. The automatic dispenser described was constructed primarily for tubing bacteriological media. It is inexpensive to build, easy to repair, and operates by hand or motor. The quantities delivered are reproduced with remarkable accuracy. A new type of valve, made from a rubber stopper and described here for the first time, has many uses in the laboratory. Any liquid or gas which does not attack rubber or glass can be dispensed in the apparatus. It can be used to circulate liquids or gases in closed systems, aerate fermentations, or as a filter pump.

Automatic cutoff for electrically heated water still. L. M. White and G. E. Secor. Indus. and Engin. Chem., Analyt. Ed. 18(5):332-333, May, 1946. A working drawing and electric circuit diagram are shown for an automatic cutoff for an electrically heated water still equipped with the constant-level device described by Holmes (C.A. 34, 64897).

\*Shutter shadow in photomicrography. F. T. Jones, Jour. Biol. Photographic Assoc. 15(4):193-194, June, 1947. Describes a source of trouble in photomicrography and explains how to avoid the difficulty under various conditions.

\*Melting point bath liquids useful up to 440°C. L. M. White. Analyt. Chem. 19(6):432-433, June 1947. Organosilicon compounds were found to be suitable for temperatures up to 440°C. A heating coil can be used in the liquids. A Hershberg melting point apparatus was modified to allow the bath liquid to be kept under an inert atmosphere.

### Analyses, Syntheses, Methods

A method of minimizing supercooling and its application in the determination of freezing points from dielectric constant measurements. T. M. Shaw. Rev. Sci. Instruments 13(1):2-5, Jan., 1942. Determination of freezing points of water in colloidal systems from dielectric constant measurements is shown to be unsatisfactory when supercooling occurs. A method is described for minimizing supercooling, which makes possible accurate determinations of freezing point. Data showing freezing points obtained for a Barnes soil at various moisture contents are included.

Determination of ammonia by a diffusion method. A. N. Prater. Indus. and Engin. Chem., Analyt. Ed. 14(9):703-705, Sept., 1942. Although several modifications of the diffusion cell technique have already appeared in the literature, it is believed that the one described is the simplest and most economical. To demonstrate its reliability, representative data are presented on the analysis of ammonium salt solutions, a meat extract, and a meat extract plus ammonium chloride solution. The data reported were obtained by the electrometric method and checked carefully by the indicator method. The latter was found satisfactory and has the advantage of being faster but may be affected by personal error.

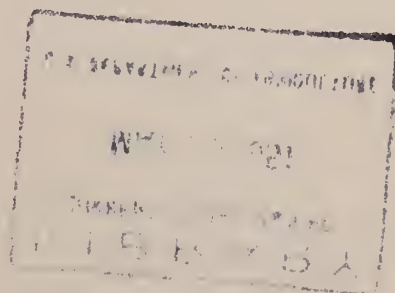
The elimination of errors due to electrode polarization in measurement of the dielectric constants of electrolytes. T. M. Shaw. Jour. Chem. Physics 10 (10): 609-617, Oct., 1942. Polarization of dielectric constant cell electrodes is an important source of error in determining dielectric properties of dilute electrolyte solutions. An investigation was made of errors resulting from electrode polarization in measurements of dielectric constants of solutions containing electrolytes. Applicability of two methods, one used by Fricke and Curtis and the other by Oncley, to eliminate these errors, was considered. There is introduced a modification of Oncley's method, which was found applicable at higher electrolyte concentrations than Oncley's method. Relative effectiveness of the modified method and that of Fricke and Curtis was tested by employing the two methods to correct experimental data obtained for aqueous solutions of glycine containing various amounts of electrolytes. Measurements were made at frequencies ranging from 10 to 5,000 kc, and at conductivities ranging from  $0.29 \times 10^{-4}$  to  $1.74 \times 10^{-4}$   $\text{ohm}^{-1} \text{cm}^{-1}$ . Corrected

values of the dielectric constant compared with known values in the literature were in good agreement. Limitations of the usefulness of the methods are discussed.

*o*-Biphenyl isocyanate, *o*-Bicyclohexyl isocyanate, *N, N'*-Di-*o*-biphenyl urea, *N, N'*-Di-*o*-bicyclohexyl urea. H. Fraenkel-Conrat and H. S. Olcott. *Jour. Amer. Chem. Soc.* 66(5):845, May, 1944. Presents methods for synthesizing the new compounds named.

The dielectric properties of beta-lactoglobulin in aqueous glycine solutions and in the liquid crystalline state. T. M. Shaw, E. F. Janson, and H. Line-weaver. *Jour. Chem. Physics* 12(11):439-448, Nov., 1944. The dielectric constant and loss factor of solutions of beta-lactoglobulin dissolved in aqueous 0.48, 1.5, and 2.5 molar glycine, and the dielectric constant of beta-lactoglobulin liquid crystals were measured for frequencies between 0.01 and 5.0 megacycles.

The composition, optical and crystallographic properties of two calcium oxalate-chloride double salts. F. T. Jones and L. M. White, *Jour. Amer. Chem. Soc.* 68(7):1339-1342, July, 1946. Reports a microscopical study of reaction of calcium oxalate with hydrochloric acid. Composition, optical and crystallographic properties and X-ray lines of a new double salt,  $\text{CaC}_2\text{O}_4\text{CaCl}_2\cdot 2\text{H}_2\text{O}$ , and those of the previously reported double salt,  $\text{CaC}_2\text{O}_4\text{CaCl}_2\cdot 7\text{H}_2\text{O}$ , were determined. The formation of these crystalline materials can be used as an aid in the microscopical identification of calcium oxalate, and to aid the phase study of this system.



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